

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2022

Volume 21, Pages 144-151

IConTES 2022: International Conference on Technology, Engineering and Science

Improving the Properties of Medium Molecular Weight Chitosan/PVA Composite Polymers via Designed ZnO Particles

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Abstract: Chitosan exhibit great potential to be a critical part of the global sustainability solution; however, its application areas are limited due to its inadequate properties for some applications. The use of chitosan is limited because of several reasons such as performance (brittleness, poor barrier properties) and processing (low thermal strength temperatures). The properties of chitosan depend on its molecular weight and there are not enough studies in the literature. The application of nanoparticles to chitosan provides new advantages both in improving the properties of chitosan and in reducing its costs. However, such nanoparticles can introduce some difficulties. Nanoparticles additives increase transparency but cause agglomerates due to the high surface/volume ratio, which reduces the local mechanical strength. Recently a novel particle technology, called as MicNo[®]. MicNo[®] particles are designed micron size hexagonal plates which are composed of primary fine particles. They only exhibit advantages of micron and nano size. For example, they exhibit superior surface coverage (up to 70% mole) with respect to spherical particles while they are transparent. Accordingly, the research goal of this study was to appraise effects of MicNo[®] and nano ZnO particles on structure development and hence properties of medium molecular weight Chitosan/PVA films. The results show that MicNo[®] particles improve UV-resistance and degree of crystallinity of the films and demonstrate great potential as new generation additive systems for biodegradable polymers to extend their application areas.

Keywords: Chitosan, Polyvinyl alcohol, Zinc oxide, UV-blocking

Introduction

The molecular weight of chitosan affects the solubility, viscosity, mechanical and antibacterial properties of the film. The solubility of chitosan depends on the pH of the solution and the molecular weight of the chitosan. Thanks to its cationic structure, chitosan can be dissolved in most dilute acids in a pH <6 environment (Knittel & Schollmeyer, 1998). As the molecular weight of chitosan increases, its solubility decreases. The high solubility of medium molecular weight chitosan appears to be attributed to reduced intermolecular interactions such as Van der Waals forces and hydrogen bonds; the lower the molecular weight, the lower the intermolecular forces of attraction (Tikhonov et al., 2006). The decrease in molecular weight causes transformation of the crystal structure, decrease in thermal stability and increase in water solubility (Qin et al., 2003). As the molecular weight increases, the viscosity of chitosan increases as the segment content in the molecular chain increases (Fox et al., 1951). As the molecular weight of chitosan decreases, the tensile strength and % elongation of the film decreases. This is due to less entanglement factor of the polymer chains. In addition, less crystallinity in the films is reported as the reason (Chen & Hwa, 1996). The molecular weight of chitosan is an effective factor in the antibacterial activity of the films. The effect of molecular weight on the antibacterial activity of chitosan is still unclear. A general conclusion could not be reached on which of the low, medium or high molecular weight chitosan exhibits the best antibacterial activity (Zheng & Zhu, 2003). Low molecular weight chitosan films show higher antibacterial activity against gram-negative bacteria species. The results of

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many studies are reported that low molecular weight chitosan can be universal antimicrobial agents against all kinds of bacteria and fungi (Gerasimenko et al., 2004). High molecular weight chitosan films show higher antibacterial activity against gram positive bacteria species. Gram-positive bacteria have an intrinsically different cell wall structure. High molecular weight chitosan surrounds bacterial cells. The polymer layer formed becomes a mechanical barrier that prevents nutrient uptake and subsequently leads to the death of the vegetative form (Fernandes et al., 2009). In order to clearly determine the factors affecting the antibacterial activity and mechanical properties of chitosan, it is necessary to examine the physicochemical properties of chitosan with different molecular weights such as crystallinity and thermal stability.

It has been proven that the addition of nanoscale additives to the chitosan matrix improves its thermal and mechanical properties, reduces moisture and gas permeability (Sorrentino et al., 2007), and achieves high antibacterial and bacteriostatic activity (Chen & Wang, 2001) as well as antioxidant properties (Heras et al., 2001). Among metal oxides, ZnO is described as a functional, strategic, promising and versatile inorganic material with a wide range of applications. Recently, ZnO has been listed as a safe material by the FDA (Food and Drug Administration, SA) (Emamifar et al., 2010). ZnO has unique optical, chemical susceptibility, semiconductor, electrical conductivity, and piezoelectric properties (Fan & Lu, 2005). It is characterized by direct broad bandgap (3.3 eV) in the near UV spectrum, high excitonic binding energy (60 meV) at room temperature, and inherent n-type electrical conductivity (Wellings et al., 2008). The wide bandgap of ZnO has significant influence on its properties such as electrical conductivity and optical absorption. Although ZnO shows slightly covalent character, it has very strong ionic bond in Zn-O. It has high stability and high heat resistance against organic materials (Padmavathy & Vijayaraghavan, 2008). In addition to its unique antibacterial and antifungal properties, ZnO nanoparticles have high catalytic and high photochemical activities. ZnO nanoparticles show biocidal effects on bacteria, fungi and viral species (Adams et al., 2006). ZnO has high optical absorption in the UVA (315-400 nm) and UVB (280-315 nm) regions, making it beneficial in antibacterial response and used as a UV protector in cosmetics (Song et al., 2011).

Since it is known that ZnO properties change depending on the shape and size of the particle, the designed plate-shaped micron particle MicNo[®] ZnO (=Micron+naNo) was used in this study. MicNo[®] ZnO has the properties of both nano and micron size. Coaxial particles with a particle size of 30-35 nm coalesced to form very thin, 2-10 μm hexagonal layered structures. The specific surface area is 23.1 m^2/g , which is larger than the specific surface area of nano-sized ZnO (14.3 m^2/g). In addition to increasing the biocompatibility feature, this provides high transparency in the visible region and high barrier feature in the UV region (Genc et al., 2018).

Studies examining the effects of the use of medium molecular weight chitosan and the morphology-size relationship of ZnO on the properties are limited in the literature. Therefore, in this study, ZnO particles in the form of nano and MicNo[®] doped with medium molecular weight Chitosan/PVA polymer and their effects on optical, mechanical and antibacterial properties were investigated.

Experimental Procedure

Materials

Chitosan (CS) with medium molecular weight and degree of deacetylation of 75% by CDH was used. In addition, PVA with average molecular weight of 190,000–205,000 Da, glacial acetic acid and glycerol with molar weight 92.09 g/mol by Sigma-Aldrich Co.LLC were utilized. MicNo[®] ZnO inorganic powders with 2-10 μm sized hexagonal platelet aggregates of 30-100 nm size particles were supplied by Entekno Materials Inc. Nano ZnO inorganic powder with <100 nm individual size particles by Sigma-Aldrich Co.LLC. was used as a reference material for bench-marking.

Preparation of CS/PVA/ZnO Composites Film

CS (300 mg) was first dissolved in 30 mL of 2% aqueous acetic acid solution and PVA (1.5 g) was dissolved in 30 mL water at 80°C. Both of the clear solutions were kept overnight while they were being stirred. After that, the PVA solution (12 mL) was added to the CS solution (8 mL) while it was being magnetically stirred to get clear solution. Then, nano ZnO or MicNo[®] ZnO (300 mg) and glycerol (0.2 mL) were added into the Chitosan/PVA solution which was at 80°C and stirred by keeping overnight. The obtained gel-like viscous solution was poured onto a plastic substrate and the films with a thickness of 200 μm were prepared by tape

casting technique and dried in air at room temperature for 24 h. In Table 1, the designations of the samples that were studied in this project, are shown.

Table 1. Used powder systems in composite films and their names

Sample No	Sample	Sample Name
1	0 (wt.%) ZnO doped Chitosan/PVA Film	ZnO-0
2	2.5 (wt.%) nano-ZnO doped Chitosan/PVA Film	ZnO-N
3	2.5 (wt.%) MicNo [®] -ZnO doped Chitosan/PVA Film	ZnO-M

Materials Characterization

X-ray diffraction (XRD) analyses were performed by D2 Phaser (Bruker, Cu $K_{\alpha}=1.54\text{\AA}$) for phase determination. The fourier transformed infrared (FTIR) spectra were collected by IRTracer-100 (Shimadzu, the wavelength range of $4000\text{--}600\text{ cm}^{-1}$). The films were placed in the holder directly in the IR laser beam. The size and morphology of the particles were examined with scanning electron microscopy (SEM EVO 50 LP, Zeiss, operating at 10 kV). The optical measurements were conducted by UV-VIS-NIR spectrophotometer (UV-3600 Plus UV-VIS-NIR, Shimadzu) in a wavelength range of 260-750 nm. The mechanical analyses were performed in a universal testing machine (INSTRON 5581) with 5 samples in each case with a dimension of $100 \times 20\text{ mm}$. Antibacterial activities of films were determined by using ISO 22196:2011 standart (Perelshtein et al., 2013). Antibacterial activity was calculated according to the ISO 22196:2011 standard, the antibacterial activity value of a surface should be $R \geq 2$.

Result and Discussion

XRD Analyses

Figure 1 shows the XRD patterns of the films evaluated in this study. There are three characteristic peak formations in Chitosan/PVA films. These are the peak at $2\theta=11.3^{\circ}$ representing the crystalline phase, the $2\theta=19.4^{\circ}$ and $2\theta=22.8^{\circ}$ peaks representing the amorphous phase. While the peaks representing the amorphous phase were obtained, the peak representing the crystal phase was not obtained. This indicates that the time elapsed during the casting process after the chitosan has dissolved in acetic acid is shorter than the time required for the crystallization of the chitosan (Azizi et al., 2014). In addition to the characteristic peaks of Chitosan/PVA chitosan films, sharp characteristic peaks of hexagonal wurtzite type crystal structure ZnO $2\theta=32.04^{\circ}$ - 34.71° - 36.54° - 47.85° - 56.94° - 63.20° - 68.32° was seen (JCPDS card no: 36-1451). These peaks belong to (100), (002), (101), (102), (110), (103) and (112) planes, respectively. This behavior indicates that the original structure of ZnO inorganic powders remains unchanged in the Chitosan/PVA matrix. The high hydroxyl group content of PVA prevents inorganic ZnO additives from agglomerating and ensures homogeneous dispersion in the Chitosan/PVA matrix (Rashmi et al., 2014) Since the specific surface area of MicNo[®] ZnO is greater than that of nano ZnO inorganic powders, it leads to an increase in the crystallinity of composite films. XRD peak intensities in MicNo[®] ZnO doped films are expected to be higher than nano ZnO doped films. However, XRD peak intensities were found to be low due to the homogeneity problem.

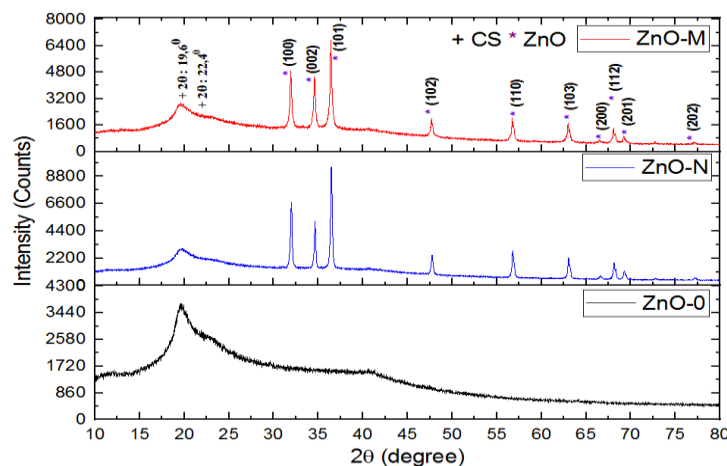


Figure 1. XRD patterns of the studied films

FTIR Analyses

The characteristic functional groups were investigated through FTIR spectra of the studied films as shown in Figure 2. The strong band between $3550\text{--}3200\text{ cm}^{-1}$ corresponds to the N-H and O-H stretches with hydrogen bonds. Absorption bands between $3000\text{--}2840\text{ cm}^{-1}$ can be attributed to C-H symmetrical and asymmetrical stresses (Biazar et al., 2015). The peak at 1543 cm^{-1} , belonging to the primary amine N-H bending, was obtained (Fernandes Queiroz et al., 2014). The reason that the intensity of the peak at 1543 cm^{-1} increases with the amount of ZnO is due to the increase in the intermolecular hydrogen bonds between -NH groups of chitosan and ZnO (Vicentini et al., 2010). The -CH_2 bending and -CH_3 symmetrical stretching are clearly visible with bands of 1458 cm^{-1} and 1371 cm^{-1} , respectively. The peak band at 1236 cm^{-1} represents the C-N stretch mode. It is seen that the absorption bands between $1155\text{--}1050\text{ cm}^{-1}$ belong to the C-O stretch (Lim & Hudson, 2004). Absorption bands between $995\text{--}875\text{ cm}^{-1}$ can be attributed to the C=C symmetrical stretch (Vino et al., 2012). With the addition of inorganic ZnO powders to the films, characteristic bands shifted to lower wavelengths and their intensity increased. The reason for this is the formation of intermolecular hydrogen bonds between PVA, Chitosan and ZnO.

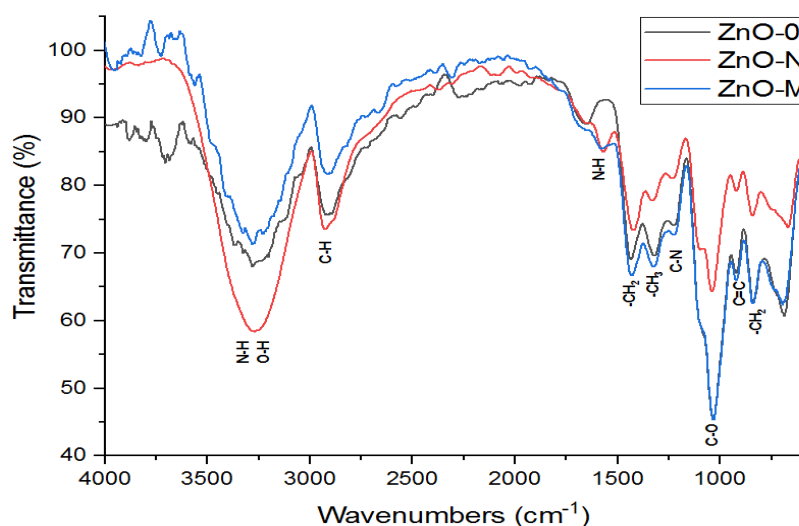


Figure 2. FTIR spectra of the studied films

SEM Analyses

Figure 3 shows SEM micrographs of the films studied in this work. Agglomerate formation is higher in MicNo[®] ZnO containing films with respect to nano ZnO containing films.

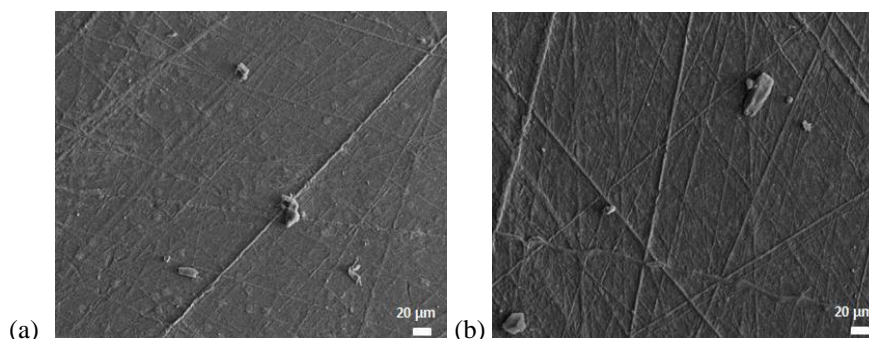


Figure 3. SEM micrographs of (a) nano ZnO and (b) MicNo[®] ZnO doped Chitosan/PVA film

UV-Visible Analyses

It was observed that the amount of absorption increased when ZnO was added in the UV region. The increase in UV absorption intensity between $355\text{--}375\text{ nm}$ indicates the presence of chromophore groups in composite films (Chouhan et al., 2015). In addition, the absorption bands shifted to higher wavelengths when ZnO was added.

The reason for this is the hydrogen bonds formed between PVA –OH groups, –OH and –NH of Chitosan and Zn ions (Kumar et al., 2014). In the UVA (315-400nm) and UVB (280-315 nm) regions, higher absorption is expected in MicNo[®] ZnO doping due to the higher surface area of MicNo[®] ZnO compared to nano ZnO doping. However, as seen in the XRD analysis, less absorption was found in the films with MicNo[®] ZnO doped due to the agglomeration problem in the films. The reason why the transparency in the visible region decreases with the addition of ZnO is that ZnO does not disperse homogeneously and forms agglomerates.

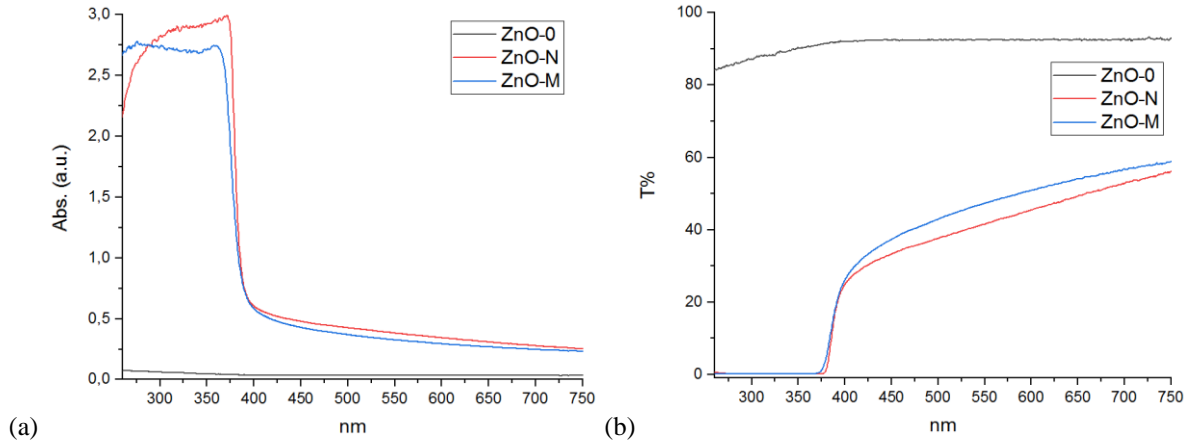


Figure 4. (a) UV-vis absorbance, (b) UV-vis transmittance spectra of the studied films

Mechanical Analyses

Table 2 shows average tensile strength (MPa) and % elongation values of the studied films in this work. The average tensile strength value of ZnO-0 was 0.58 MPa and the average % elongation amount was 16.42 %. An increase in tensile strength and a decrease in % elongation were observed with the addition of inorganic ZnO particles into the film. Whereas the tensile strength and the elongation values were 0.90 MPa and 10.20 %, respectively, for the nano ZnO containing system, they were 1.52 MPa and 13.33 %, respectively, for the MicNo[®] ZnO containing system. The reason for this change in mechanical strength is probably due to hydrogen bonds formed between PVA, Chitosan and Zn²⁺ ions. In addition, this interaction restricts the movement of molecular chain segments, which causes a decrease in elongation (Yin et al., 2018).

Table 2 Average tensile strength (MPa) and % elongation values of the studied films

Sample Name	Average Tensile Strength (MPa)	Average Elongation (%)
ZnO-0	0.58	16.42
ZnO-N	0.90	10.20
ZnO-M	1.52	13.33

Antibacterial Analyses

In Table 3, the antibacterial activity (R) values against S.Aureus and E.Coli of the studied films are shown. ZnO-0 and ZnO-N films do not exhibit adequate antibacterial effect. With the addition of MicNo[®] ZnO particles with high antibacterial activity into the films, the R values became compatible with the ISO 22196:2011 standard ($R \geq 2$). As the specific surface area of ZnO increases, the contact surface area/volume between microorganisms and the particles increases as in the MicNo[®] case and this interaction makes the adsorption of ZnO particles onto the microbial cell walls mechanism dominant (Demirel et al., 2018). Therefore, the R values of the MicNo[®] ZnO doped films were higher than the nano ZnO doped films.

Table 3 The antibacterial activity (R) values against S.Aureus and E.Coli of the studied films

Sample Name	S.Aureus (R)	E.Coli (R)
ZnO-0	0.08	0.30
ZnO-N	1.38	1.70
ZnO-M	2.47	2.57

Inhibition experiment (Figure 5) was applied with agar diffusion method. The occurrence of the inhibition zone indicates that the tested ZnO powder systems have different levels of antibacterial activities on bacteria (Demirel et al., 2018). ZnO-M films appear to inhibit S.Aureus (1-2 mm).

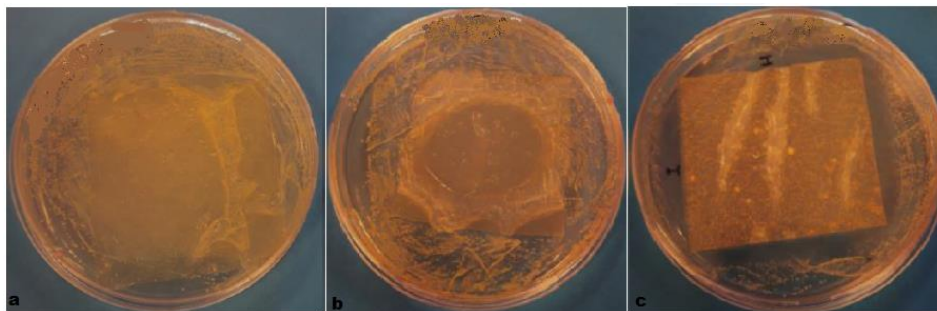


Figure 5. Inhibition zone thicknesses (mm) against S.Aureus of (a) ZnO-0, (b) ZnO-N, (c) ZnO-M

Conclusion

The changes in the film properties were investigated with ZnO doping with different size & morphology (nano-MicNo[®]) to the medium molecular weight Chitosan/PVA matrix. With ZnO doping, the degree of crystallinity and UV-blocking capacity of the films increase. Since the specific surface area of MicNo[®] sized ZnO doped films is higher than nano sized ZnO, this increase is expected to be more effective. However, due to the use of 2% (v/v) glycerol, a tendency to agglomerate was observed in the films, as supported by SEM analysis, and the homogeneous distribution of inorganic powders along the film surface was not realized. According to the mechanical strength analysis, it was observed that there was an increase in tensile strength and a decrease in % elongation with inorganic ZnO doping of the medium molecular weight Chitosan/PVA films. According to the antibacterial analysis, no bacterial growth was observed in the ZnO-M films, and a bacteriocidal effect was found.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Acknowledgements or Notes

* This article was presented as a poster presentation at the International Conference on Technology, Engineering and Science (www.icontes.net) conference held in Antalya/Turkey on November 16-19, 2022.

*This research was supported financially by the Eskisehir Technical University Scientific Research Projects Commission (Project number: 20DRP036). The authors would like to thank Prof. Dr. Rasime Demirel for her contributions for the antibacterial tests.

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To cite this article:

Kose, Y., & Suvaci, E., (2022). Improving the properties of medium molecular weight chitosan/pva composite polymers via designed ZnO particles. *The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM)*, 21, 144-151.