



Synthesis and Evaluation of Mechanical Property of Chitosan/PVP Blend Through Nanoindentation-A Nanoscale Study

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Abstract

Biocompatible chitosan/PVP blends of different weight ratios were prepared by the solution casting method. The blends were characterized for morphology, structural, functional group, and thermal analysis. SEM images revealed the homogeneous mixing of both polymers at a certain weight ratio. XRD results show that the crystallinity of the blend decreases with an increase in PVP concentration. The blends were also characterized for the evaluation of physical properties like contact angle and water vapor transmission rate. A nanoindentation test was performed to characterize the mechanical strength, elastic modulus as well as hardness of these films. The water vapor transmission rate and hydrophilicity of the blend significantly decrease with an increase in the concentration of PVP. The blend with CH/PVP (75:25) showed the most satisfactory mechanical strength as compared to all tested ratios. Furthermore, these blends had good transparency and thermal stability that can be the alternative of petroleum-based synthetic products.

Keywords Chitosan/PVP blends · Solution casting · Crystallinity · Nanoindentation

Introduction

Since the 1980's an excessive use of petrochemical-based polymers, have made them a major source of pollutants after use due to their non biodegradability. So, researchers and govt. agencies show greater concern about the environmental problem caused by the use of non-biodegradable plastic material for various purposes [1–3]. In this regard, the use of natural resources is being explored to produce biodegradable material instead of non-biodegradable petrochemical-based counterparts [3]. After the use of biopolymers, it is desired to be degraded in a reasonable period without affecting the environment [4, 5]. Biopolymers such as starch [6], chitosan [7], cellulose [8] have been considered as an effective alternative of conventionally used polymers due to its abundant, renewable, environmentally friendly, and biodegradability.

Chitosan is a natural biopolymer, polycationic, deacetylated derivative of chitin, a major component of crustacean

outer skeletons. It is a linear polysaccharide made up of repeating units of β -(1–4)-linked 2-acetamido-2-deoxy-D-glucopyranose units [9, 10]. Owing to its biodegradable, non-toxic, excellent film-forming ability, strong antimicrobial and antifungal activity, chitosan based material plays an important role in biotechnology, pharmaceuticals, biomedicine, cosmetic, wastewater treatment, food science, and packaging, etc. [11–13]. Chitosan has been widely used to prepare active biodegradable film due to its good thermal and chemical resistant property and extend the shelf life of food and prevent contamination. Despite several advantages and unique properties chitosan films have some disadvantages associated like a poor barrier to water vapor, gas, poor mechanical, and thermal property limit its use for various applications [14, 15]. Therefore, to get the desired results polymer blending is the most efficient way to get the desired characteristics in the film. Various synthetic and a natural polymer such as poly *N*-vinyl pyrrolidone [16], polyethylene oxide [17], starch [18], cellulose [19], collagen [20], carboxymethyl cellulose [21], and cellulose acetate [22] are used to blend with chitosan to enhance the mechanical and barrier property of it.

Poly (vinylpyrrolidone) (PVP) is a synthetic polymer, known for its biocompatibility, non-toxicity, and hydrophilic properties, frequently used in controlled drug release,

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wound dressings, and tissue engineering [14, 23]. Also, PVP is a water-soluble polymer that has beneficial effects on absorbency, viscosity, solubilization, condensation, and is capable of forming blend with chitosan. PVP can form hydrogen bonding with chitosan by bond formation between the amino and hydroxyl group of the chitosan and carbonyl group of PVP [24, 25]. The blend of chitosan and PVP has been widely used in biotechnology and chemical technology. Aldana et al. reported the fabrication of chitosan/genipin/PVP film for the controlled release of drugs [26]. Smitha et al. reported the formation of PVP/chitosan blends as membranes for methanol fuel cell applications. and study the methanol permeability [27]. However, in these research papers transparency, nanoindentation, and water vapor permeability of the film were not evaluated.

It is important to study the nano and micro scale mechanical properties of chitosan and chitosan/PVP films since they have been widely used for biomedical applications. At very small scales the standard methods do not help in determining the mechanical properties of the material. The nanoindentation technique is a very accurate technique utilized for the measurement of mechanical properties of materials at a small length scale with high precision in load and displacement. For soft materials, high loads are avoided to prevent deformation; the full range of force from micro newton to millinewton can be applied using nanoindentation. Hardness (H), elastic modulus (E), and other mechanical properties can be determined using this technique.

The current research work aims to blend chitosan and PVP, at the different ratios for successful production of film containing the properties of both the polymers as to achieve the desired results. These films were characterized regarding their morphology, structural, optical properties, thermal stability, mechanical performance, water vapor permeability, to anticipate their potential application for food packaging as well as biomedical film.

Experimental

Materials

Chitosan (low molecular weight), Polyvinylpyrrolidone (PVP) (K-30, $M_w = 40$ kDa), acetic acid of analytical grade were procured from fisher scientific. All chemicals were used without further purification. Double distilled water was used throughout the experiment.

Preparation of Chitosan/PVP Blend

The solution casting method was used to prepare the blend of different concentrations of chitosan and PVP. Figure 1a shows the scheme to prepare the chitosan/PVP blend and

b shows the proposed reaction mechanism to prepare the chitosan/PVP blend. Different concentration of chitosan was dissolved in 1 N acetic acid solution for 5 h at room temperature with continuous stirring for complete mixing. Afterward, the various quantity of PVP was added with constant stirring for 24 h to get a homogeneous solution. The resulting solution was then centrifuged at 3000 rpm for 15 min to remove the air bubbles and to remove the undissolved particles. The solution was then cast onto a glass petri dish and dried at 50 °C in a hot air oven for 12 h. The dried films were peeled from the petri dishes and stored in polybags in a desiccator for future use. The chitosan/PVP blend of 100:0, 75:25, 50:50, 25:75 has been prepared.

Characterizations

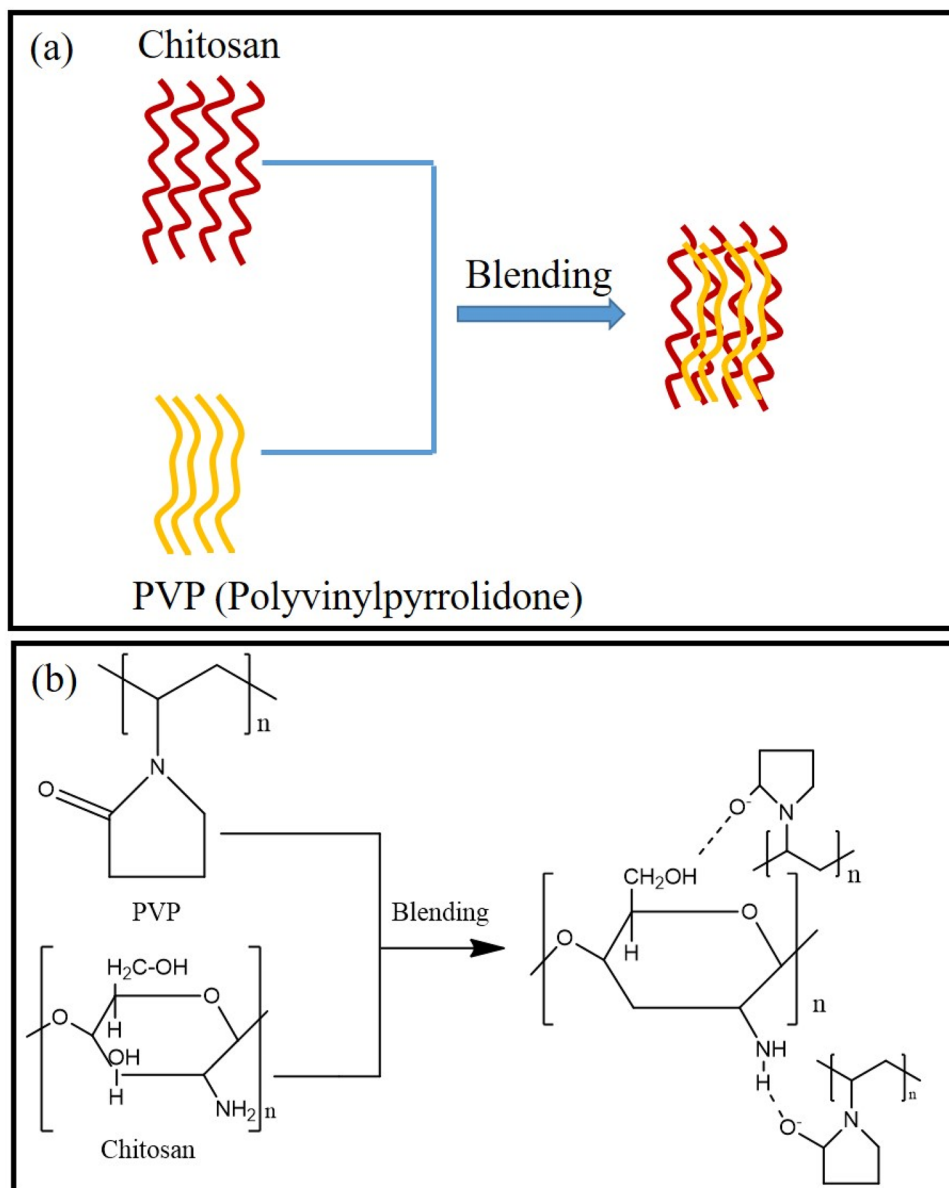
Surface morphology of chitosan/PVP blended films was examined using scanning electron microscopy (Model: EVO18 Make: ZEISS). X-ray diffraction pattern of each of chitosan/PVP blend was measured using X-ray diffractometer (Model: Miniflex PDXL, Make: Rigaku) to evaluate crystallinity in the film. The functional group analysis of prepared blends was measured using an attenuated total reflectance fourier transform (ATR-FTIR) spectrometer (Model: Nicolet iS5N FT-NIR Spectrometer, Model: Thermo Scientific) in the wavelength range of 500 cm^{-1} to 4000 cm^{-1} . The transmittance spectra of the chitosan/PVP blend were scanned from 200 to 800 nm wavelength using a UV-Vis spectrophotometer (Model: double beam spectrophotometer U-2900/2910, Make: HITACHI). The hydrophilicity of prepared blends was measured using a drop shape analyzer (Model: DSA25 Make: Kruss). The thermal stability of films was measured using the TGA instrument (TGA Q50/Q500, Make: TA instruments) in the temperature range of 30°C to 600°C under continuous nitrogen flow. The water vapor transmission rate of blends was measured by the gravimetric technique according to the ASTM E 96/E 96M-05 (water method) at the temperature of 32 ± 2 °C and relative humidity of $50 \pm 2\%$. The nanoindentation experiment on the blends was performed using anton parr nano-indentation system at room temperature.

Result and Discussion

Scanning Electron Microscopy

To determine surface morphology, surface characteristics, SEM image of the CH/PVP blends were evaluated. Figure 2a shows the SEM image of pristine chitosan film. Figure 2b, c shows the smooth and even surface of blends (75:25, 50:50) indicating homogeneous blending between chitosan and polyvinylpyrrolidone. The smooth and even surface of the

Fig. 1 a Schematic image to prepare the blend of chitosan and PVP (b), Proposed reaction mechanism to prepare chitosan/PVP blend



blend is might be due to the semicrystalline nature of the polymer. However, 2d shows phase separation might be due to the poor compatibility between chitosan and PVP at a certain weight ratio [14, 28].

X-Ray Diffraction

X-ray diffraction was performed to determine the crystalline structure of the chitosan/PVP blend (Fig. 3). XRD peak of pristine chitosan shows its characteristics peak at $2\theta = 9.16^\circ, 11.30^\circ, 16.09^\circ, 18.13^\circ, 22.94^\circ$ indicating semi-crystalline nature. Whilst with the increase in the concentration of PVP the peaks become broader. In particular, till chitosan/PVP (50/50 mass/mass) blend shows semicrystalline nature after that blend becomes more amorphous. The intensity of the

peak also decreased with an increase in the concentration of PVP due to enhancing complexation between chitosan and PVP [29]. The crystallinity of blend with low PVP concentration can explain the enhanced mechanical properties exhibited by the blends, as well as the high glass transition temperature (T_g). It is a well-known fact that the degree of crystallinity has a positive effect on both of them.

Fourier Transform Infrared Spectroscopy

FTIR spectra of chitosan and its blend with PVP at different ration is shown in Fig. 4. Chitosan and PVP are strongly interacted with each other and form a homogeneous phase because of strong hydrogen bonding. In the chitosan spectra, the peak at 3424 cm^{-1} and 3267 cm^{-1} are attributed to the

Fig. 2 SEM image of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

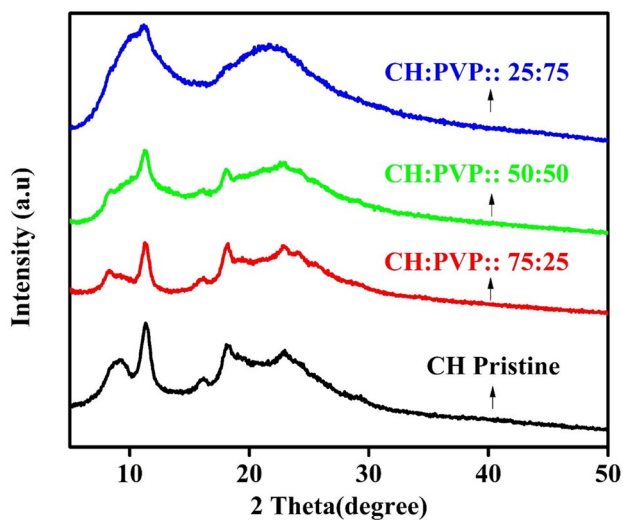
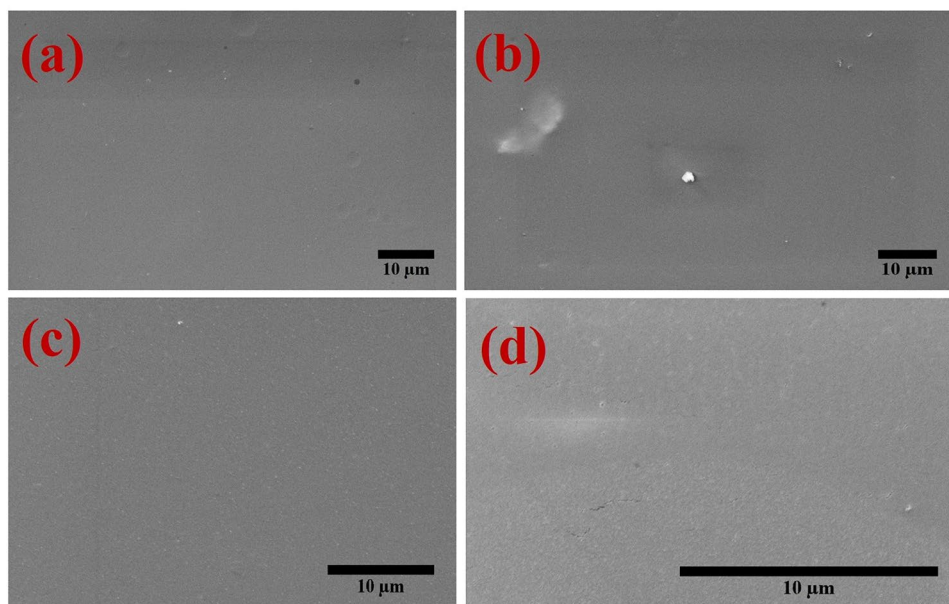


Fig. 3 XRD pattern of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

OH and NH_2 group, respectively. The peak at 2921 cm^{-1} and 2874 cm^{-1} corresponds to the asymmetric and symmetric stretching of CH_2 group vibration. 1646 cm^{-1} and 1537 cm^{-1} corresponds to the C–O stretch of an acetyl group, N–H bending vibration of secondary amide. The presence of sharp peak at 1298 cm^{-1} in Chitosan/PVP (25:75, 50:50) spectra corresponds to the stretching vibration of C–N (because of pyrrolidone structure). The presence of peak at 1537 cm^{-1} and 1298 cm^{-1} in Chitosan/PVP blend confirmed the homogeneous blending of chitosan and PVP. The peak position at 3434 cm^{-1} in pristine chitosan is shifted to a lower frequency in the chitosan/PVP blend confirming the interaction of chitosan and PVP by means of intermolecular

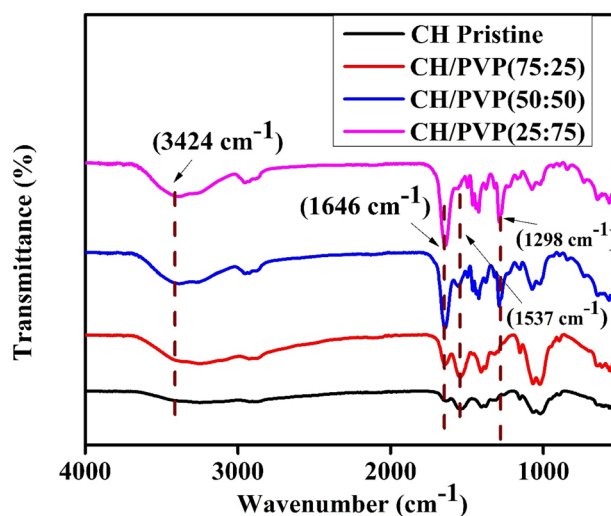


Fig. 4 FTIR spectra of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

hydrogen bonding. Moreover, shifting of carbonyl bond from 1646 cm^{-1} to lower frequency confirming the interaction between chitosan and PVP [15, 24, 28].

UV–Vis Spectroscopy

UV–Vis spectra of all blends are shown in Fig. 5. Pristine chitosan film shows the optical transmittance of 60% whereas, with an increase in the concentration of PVP in chitosan/PVP blend transmittance increased up to 85% which indicated the homogeneous mixing of chitosan and PVP. The highly transparent film is suitable for transparent packaging as well as biomedical applications. Figure 6 provides camera

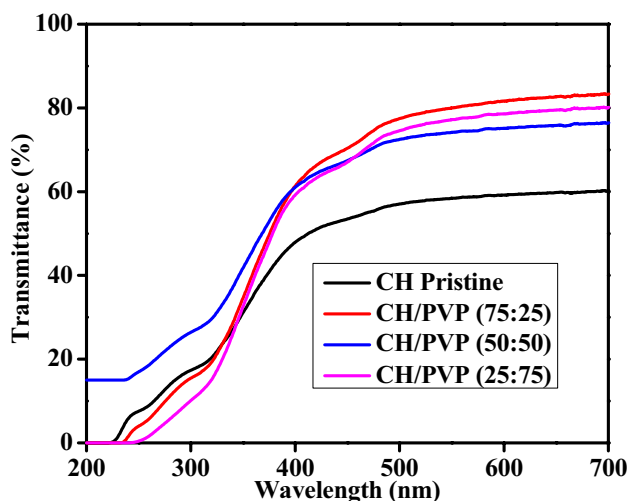


Fig. 5 UV-Vis spectra of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

images of CH/PVP blends. It can be seen from images that the blends have excellent transparency. The highly transparent film is suitable for various applications.

Contact Angle

Figure 7 shows the contact angle of pristine chitosan and blend of different concentrations of chitosan and PVP. It shows that the contact angle of pristine chitosan is 79.3° whilst with the increase in the concentration of PVP in the blend the contact angle decreases. The hydrophilicity of PVP is mainly due to hydrophilic amino and carboxyl groups and hence results in a decrease in the contact angle of the film when blended with chitosan [14, 30].

Water Vapor Transmission Rate

The water vapor transmission rate is the most important feature of the blend for its application in the packaging industry. It works on the principle of the diffusion process

in which water vapor condenses and then liquid water diffuse through the film. Figure 8 shows the water vapor transmission rate of pristine chitosan and blend of different concentrations. Due to the hydrophilic nature of PVP water vapor transmission of blend with increasing concentration of PVP shows a significant increase in water vapor transmission. However, the blend with CH/PVP (75:25) shows lesser water vapor transmission compared to other compositions of chitosan, and PVP might be due to the strong intermolecular force of interactions between chitosan and PVP molecules results in the shorter intermolecular distance forming more compact films [24].

Thermogravimetric Analysis

The thermal stability of the pristine chitosan and CH/PVP blend of different compositions was studied by mean integral (TGA) and results are shown in Fig. 9. The results from the TGA shows the weight loss vs temperature for the prepared blend. Pristine chitosan and blend of CH/PVP shows an initial weight loss of 15% in the region of $50\text{--}180^\circ\text{C}$ due to the evaporation of absorbed water molecule. The initial onset temperature (T_{onset}) for degradation was observed at 223.57°C with 18% weight loss and offset temperature (T_{offset}) was about 500°C . Pristine chitosan shows degradation at $180\text{--}400^\circ\text{C}$ due to the depolymerization of the chitosan backbone through cleavage of glycosidic linkages and above 400°C due to the decomposition of carbonaceous matter [15]. The blend of CH/PVP with different concentrations show degradation in the region of $180\text{--}390^\circ\text{C}$ due to the degradation of chitosan and $390\text{--}480^\circ\text{C}$ due to the decomposition of PVP. At 500°C , the residue of the CH/PVP blend is less than pristine chitosan due to the fastest degradation of PVP. In CH/PVP (75:25) blend no residue is left at the temperature of 500°C because of the fastest degradation of PVP [28].

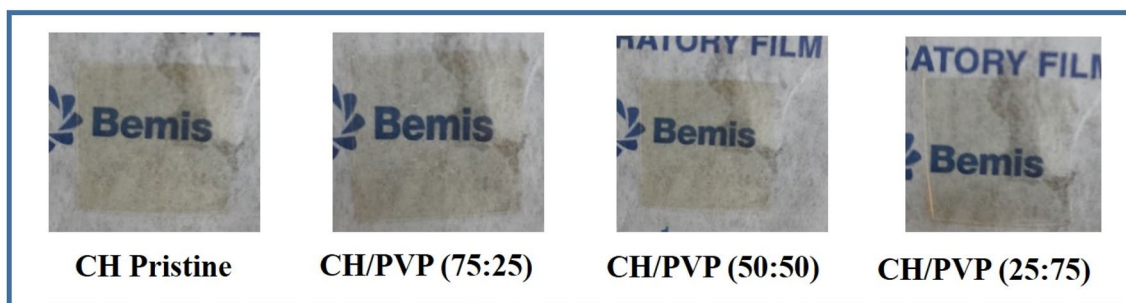


Fig. 6 Camera images of the of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

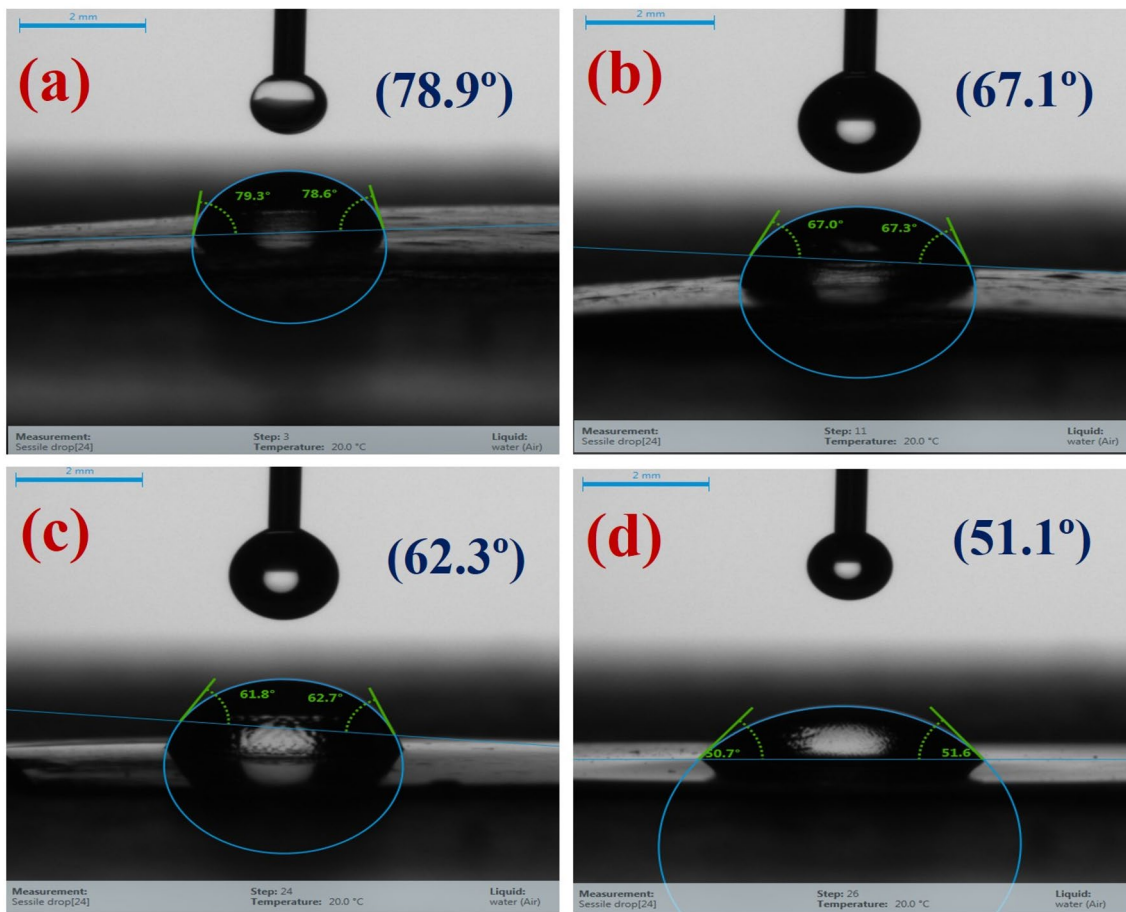


Fig. 7 Contact angle of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

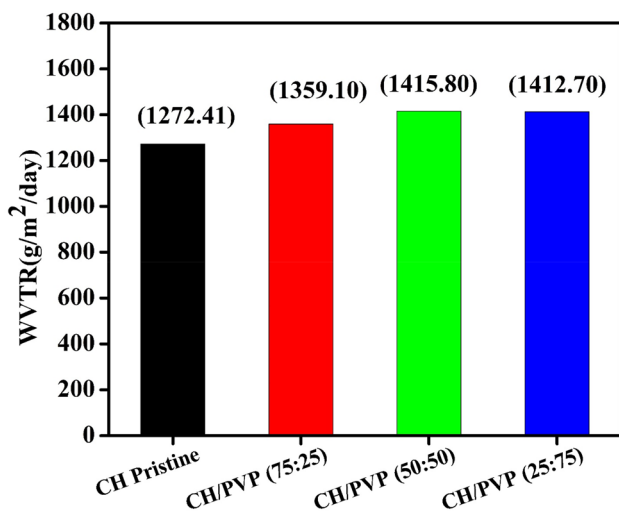


Fig. 8 WVTR of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

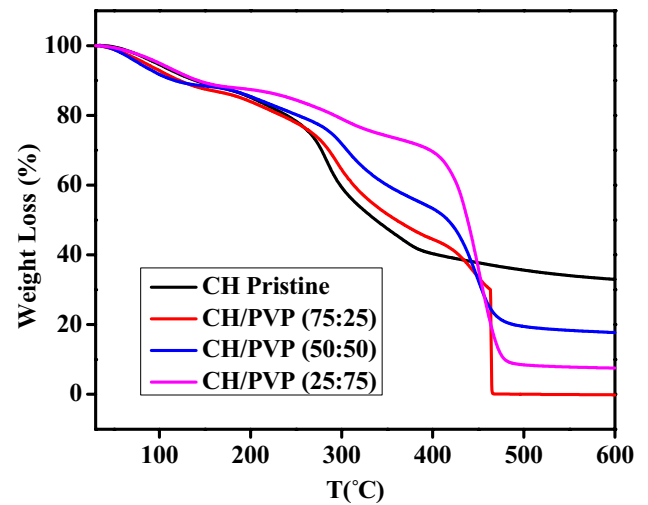


Fig. 9 TGA curve of chitosan/PVP blend (100:0, 75:25, 50:50, 25:75)

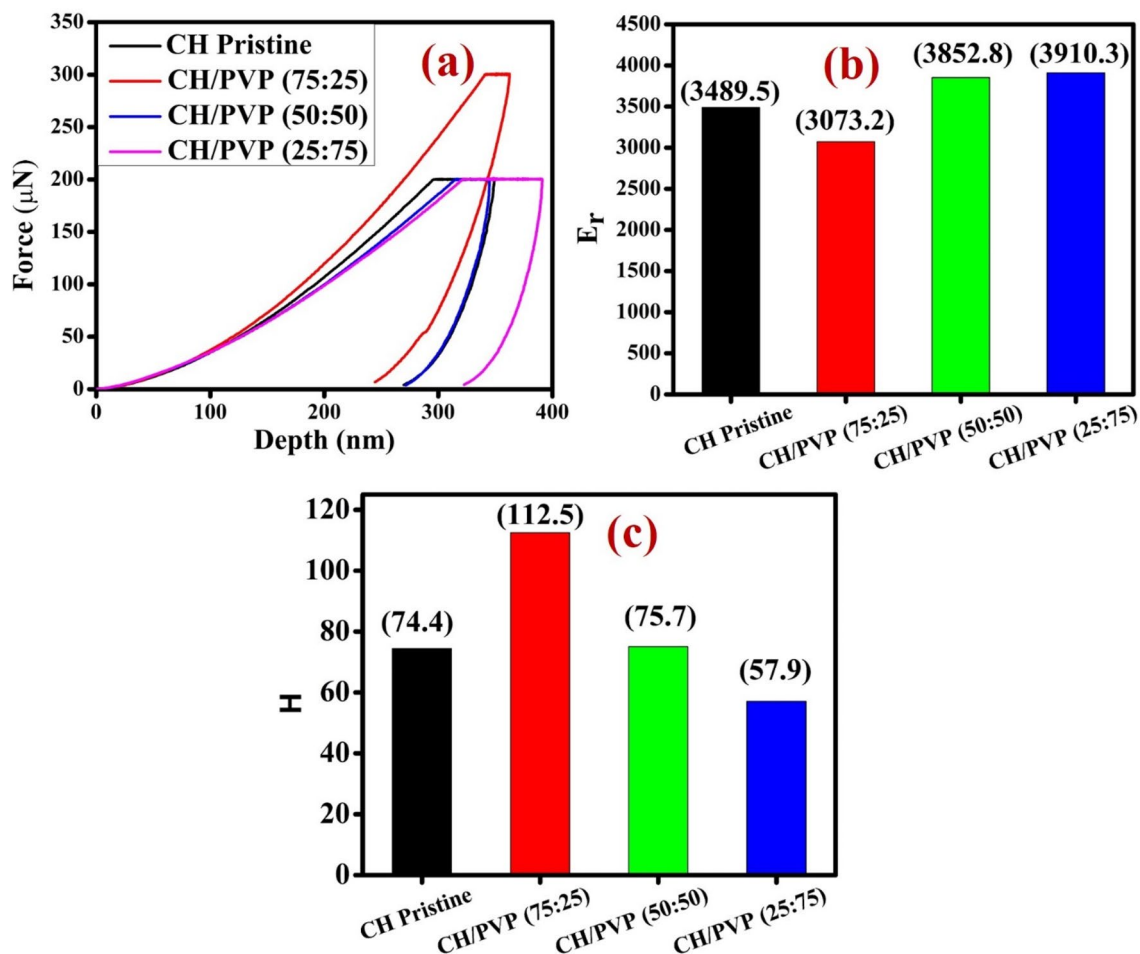


Fig. 10 Nanoindentation of CH/PVP blend (100:0, 75:25, 50:50, 25:75)

Nanoindentation

Figure 10 shows the graphs for quasi-static nanoindentation studies on the CH/PVP blend of 100:0, 75:25, 50:50, 25:75 ratios (using a Berkovich tip).

Figure 10a shows Force–penetration depth (F–d) curves for CH/PVP Blends. The area for indentation testing was carefully chosen using an optical microscope and indentation was done in the load control mode. The effect of surface roughness was avoided by fixing the contact load as 30 μN. F–d curves show that pristine chitosan film has some amount of plastic deformation which increases with an increase in the concentration of PVP in the blend. However, the sample with 75:25 ratio shows a lesser plastic area in comparison with the pristine chitosan sample also the holding capacity of the sample shows an increase with the increase in PVP concentration.

The elastic modulus (E_r) and hardness (H) Fig. 10b, c for CH/PVP blend of 100:0, 75:25, 50:50, 25:75 ratios samples were calculated using parameters obtained from

indentation force–displacement curves. The properties of samples were calculated using sneddon's elastic contact theory, the reduced modulus was calculated using the formula (1)

$$E_r \frac{S}{2\beta} \sqrt{\pi/A_c} \quad (1)$$

where S ($S = dP/dh$) is the unloading stiffness, which is calculated using the slope of the unloading curve of F–d data. β depends on the geometry of the indenter, which is called the correction factor. For our berkovich tip $\beta = 1.034$. For berkovich indenter projected contact area (A_c) depend on contact depth (h_c) using the formula (2)

$$A_c = 24.5 hc^2 \quad (2)$$

$$hc = h_{max} - \epsilon P_{max}/S \quad (3)$$

where h_{max} is maximum displacement, and constant ϵ is taken as 0.75 for berkovich indenter.

The elastic modulus of the sample was calculated using reduced modulus E_r in the formula

$$\frac{1}{E_i} = \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_i^2}{E_i} \quad (4)$$

where E is elastic modulus, ν is poison ratio, and subscript s is for sample, and i is for indenter.

In our study, the elastic modulus of berkovich indenter E_i is 1141.00 GPa, and the poison ratio is 0.07. By using the contact area, the hardness of the sample can be obtained using an Eq. (5)

$$H = \frac{P_{\max}}{A_c} \quad (5)$$

Pristine chitosan shows the hardness of 74.41 MPa. The blend with chitosan/PVP (75:25) shows an increased hardness of 112.5 MPa compared to pristine chitosan film. The increase in hardness is probably due to the strong interaction between chitosan and PVP through intermolecular hydrogen bonding. Whilst with an increase in the concentration of PVP into chitosan PVP blend hardness decreases. This could be due to the rigid and fragile nature of PVP and the added PVP destroyed the continuity of chitosan molecules, resulting in a decrease in the blended polymer strength [27, 28]

Conclusions

High-performance novel polymer blends were successfully prepared by solution casting of chitosan and polyvinylpyrrolidone (PVP) at different weight ratios. FT-IR analysis of blended polymers confirmed the compatibility and miscibility of both the polymers. The strong intermolecular hydrogen bonding interaction between amino and hydroxyl group of chitosan with the carbonyl group of PVP, results in the formation of a new biocompatible homogeneous blend. Contact angle measurement revealed that with the increase in the concentration of PVP in the blend the hydrophilicity of blends increased. TGA analysis shows that the blends are thermally stable. The water vapor transmission rate of blends increased with an increase in the concentration of PVP. Nanoindentation study also reveals that the mechanical strength shows a decrease in hardness and an increase in elastic modulus as the concentration of PVP increases, with an exception for the sample having a blend ratio of 75:25, wherein an increase in hardness but a decrease in elastic modulus is observed. An increase in holding time and plastic area is also observed with increasing concentration of PVP. Such results could be explained due to structural modification in the synthesized composites due to interpolymeric complexes with physical linkage with the participation of PVP. Moreover, the blends are highly transparent and its

transparency increases with an increase in the concentration of PVP that can be utilized for transparent packaging as well as for the biomedical applications.

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Declarations

Conflict of interest The authors declared no conflict of interest.

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