

Article

Development of UV-Resistant Chitosan/Starch Biofilms Reinforced with Chitosan Nanoparticles for Sustainable Packaging

Mithilesh Yadav ^{1,*} , Priyanka Maurya ¹, Pranjyan Dash ², Akash Seth ¹, Deepak Yadav ³ , Monika Jain ⁴, Jasaswini Tripathy ⁵ , Arpit Sand ⁶ , Prakash Chandra ⁷ and Pradeep Kumar Panda ^{8,*}

- ¹ Department of Chemistry, Prof. Rajendra Singh (Rajju Bhaiya) Institute of Physical Sciences for Study and Research, Veer Bahadur Singh Purvanchal University, Jaunpur 222003, India; pm99342@gmail.com (P.M.); as9670157878@gmail.com (A.S.)
 - ² Department of Chemical Engineering and Biotechnology, National Taipei University of Technology (Taipei Tech), Taipei 10608, Taiwan; pranjyandash@gmail.com
 - ³ Department of Chemistry, IIT, Guwahati 781039, India; deepakbhu15798@gmail.com
 - ⁴ Department of Natural Resource Management, College of Forestry, Banda University of Agriculture & Technology, Banda 210001, India
 - ⁵ Department of Chemistry, School of Applied Sciences, KIIT University, Bhubaneswar 751024, India; jasaswinitripathy@gmail.com
 - ⁶ Department of Sciences (Chemistry), Manav Rachna University, Faridabad 121004, India; sand.arpit@gmail.com
 - ⁷ Department of Chemistry, Bundelkhand University, Jhansi 284128, India; prakash@bujhansi.ac.in
 - ⁸ Department of Chemical Engineering and Materials Science, Yuan Ze University, Taoyuan 32003, Taiwan
- * Correspondence: mithileshvbspu2019@gmail.com (M.Y.); rkpanda277@gmail.com (P.K.P.); Tel.: +91-8738045471 (M.Y.)

Abstract

The fabrication of sustainable packaging films based on chitosan/starch (CTS/Starch) blends, reinforced with Chitosan Nanoparticles (CNPs), was achieved *via* the casting blend technique. This research explored the impact of varying CNPs loading on critical physicochemical properties, including water vapor permeation (WVP), thermal stability, and mechanical strength. To elucidate the structural and chemical complexities of the blend films, surface morphology was investigated *via* Scanning Electron Microscopy (SEM), internal architecture was visualized using Transmission Electron Microscopy (TEM), and molecular interactions were probed through Fourier Transform Infrared (FTIR) spectroscopy. The reduction in WVP from 6.18 ± 0.54 to $5.38 \pm 0.93 \text{ g.m}^{-1}.\text{s}^{-1}.\text{pa}^{-1}$, equilibrium moisture content (EMC) from $16.52 \pm 1.03\%$ to $12.5 \pm 1.05\%$, and water absorbency (WA) from $340 \pm 1.63\%$ to $88.65 \pm 1.12\%$ in CTS/Starch blend films demonstrated loaded with (0–8 wt%) CNPs loading. Concurrently, films with 2–8 wt% CNP loading exhibited an increase in opacity from $2.38 \pm 1.01 \text{ mm}^{-1}$ to $4.83 \pm 0.83 \text{ mm}^{-1}$, accompanied by a decrease in transmittance from $89.20 \pm 0.50\%$ to $79.70 \pm 1.20\%$. These findings collectively indicated that the CNP-incorporated chitosan/starch composites offer enhanced ultraviolet light shielding and improved water barrier capabilities compared to the non-reinforced chitosan/starch films, underscoring their promising utility in food and pharmaceutical packaging applications.

Keywords: chitosan; starch; chitosan nanoparticles; FTIR; mechanical properties



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1. Introduction

At every stage of the food supply journey—spanning harvest, storage, transit, processing, retail, dining, and eventual consumption—food encounters a spectrum of natural and artificial light sources [1]. This continuous light exposure, particularly to UV radiation, deteriorates food quality through photolysis and photooxidation. It is of significant concern as it rapidly degrades the sensory and nutritional properties of both raw and packaged food, altering its external and internal characteristics and representing a major cause of spoilage. Consequently, innovative solutions to prevent food deterioration are urgently needed, and food packaging serves this purpose by extending shelf life through the reduction in physicochemical changes in color, flavor, weight, bioavailability, texture, and moisture content. It also ensures safety against chemical and microbiological contamination while enhancing ergonomics, flexibility, and ease of handling, providing protection from external factors like UV radiation and humidity [2].

Packaging materials such as paper, brown glass bottles, aluminum foil, and paper-board effectively block ultraviolet (UV) radiation, thereby protecting contents from photooxidative degradation. However, transparent packaging is often preferred for its ability to enhance product visibility, consumer appeal, and brand differentiation [3]. The development of high-performance UV-blocking packaging materials is essential for maintaining food quality, with petroleum-based plastics and their blends presently representing the predominant materials employed in the food packaging sector. The widespread use of plastic in food packaging has driven its substantial growth over the past few decades due to its advantageous combination of mechanical stability, affordability, vapor and oxygen barrier properties, and transparency. However, the increasing reliance on plastics has raised environmental concerns and highlighted the depletion of petroleum resources, leading to a growing demand for biodegradable alternatives [4,5]. Research efforts are increasingly being directed towards the identification, characterization, and application of novel biopolymer-based UV protective films derived from natural sources, such as chitosan, cellulose, starch, gelatin, carrageenan, and alginic acid [2,6–8].

Starch, a biopolymer composed of amylose and amylopectin linked by α -D-glycosidic bonds [9], exhibits exceptional mechanical and film-forming qualities, making it appealing for edible packaging [10,11]. However, its hydrophilic nature limits broader application due to high water sensitivity and poor moisture barrier properties [11]. Chitin—the structural polysaccharide in arthropod exoskeletons and fungal cell walls—is widely recognized as the second most abundant natural polysaccharide after cellulose [12]. Chitosan (CTS) is the deacetylated derivative of chitin and is extensively studied for its biocompatibility and functional versatility [13]. While pure chitosan films exhibit functional properties like gas-barrier performance and antibacterial activity, they often lack the mechanical robustness required for industrial use [14].

To overcome the limitations of individual biopolymers, the incorporation of chitosan nanoparticles (CNPs) has emerged as a transformative strategy. CNPs are typically synthesized via ionic gelation—a method involving the electrostatic interaction between the positively charged amino groups of chitosan and negatively charged polyanions like tripolyphosphate (TPP)—resulting in particles with high surface-to-volume ratios. Previous studies have demonstrated that CNPs significantly reinforce diverse polymer matrices; for instance, they have been successfully incorporated into biopolymers such as gellan gum, carboxymethylcellulose (CMC) to increase tensile strength [15], used in starch-polyvinyl alcohol blends to enhance structural durability [16], and added to gellan gum films to improve moisture resistance [17]. These applications highlight the versatility of CNPs in developing active packaging for dairy, meat, and horticultural products.

Despite the documented success of CNPs in various systems, their synergistic interaction within a combined (CTS/Starch) binary matrix remains significantly under-explored. Most existing literature focuses on starch-nanocellulose or chitosan-metal oxide composites, often overlooking how CNPs can specifically bridge the compatibility gap between chitosan and starch to create a more cohesive crystalline network. This study is guided by the research hypothesis that the localized crystalline domains formed by CNPs will simultaneously act as a “tortuous path” for water vapor and a “nano-shield” against UV radiation without compromising the films’ rapid biodegradability in soil.

The novelty of this work lies in the systematic optimization of CNP loading (from 2% to 8%) within a CTS/Starch blend to produce a high-performance nanocomposite that outperforms previously developed biopolymer films in terms of UV opacity and mechanical resilience. This work provides the first comprehensive performance profile—encompassing structural (FTIR/XRD), morphological (SEM/TEM), barrier, thermal, and environmental soil burial properties—of this specific ternary system, establishing it as a superior, eco-friendly alternative to synthetic UV-protective packaging.

2. Materials and Methods

2.1. Materials

Chitosan (85–90% deacetylated, molecular weight ≈ 350 kDa) was purchased from SRL Chemicals (Mumbai, India) and exhibited a moisture content of approximately $8.2 \pm 0.5\%$, as determined by oven drying at 105°C until constant weight. Soluble potato starch (amylose content $\sim 20\text{--}25\%$, amylopectin content $\sim 75\text{--}80\%$, molecular weight $10^6\text{--}10^7$ Da, moisture content $11.4 \pm 0.7\%$) was obtained from CDH Fine Chemicals (New Delhi, India). Acetic acid (glacial), tripolyphosphate, sodium chloride, glycerol, and ethanol (analytical grade, 95%) were procured from CDH Fine Chemicals (New Delhi, India). All the materials were used as received without further purification.

2.2. Synthesis of CNPs

Chitosan nanoparticles (CNPs) were synthesized *via* a modified ionic-gelation technique, employing tripolyphosphate (TPP) as the cross-linking agent [18,19]. The chitosan solution was prepared in accordance with the procedure outlined in a previous report [17]. For nanoparticle formation, a 1% (*w/v*) aqueous TPP solution was added dropwise to the 200 mL of chitosan solution (2.0% *w/v*, prepared by dissolving chitosan in 2% *v/v* acetic acid to obtain chitosan acetate salt at a rate of 1 mL min^{-1} using a syringe pump with vigorous stirring at ambient temperature. The suspension was sonicated in a Labman Ultrasonic Bath Sonicator (Model LM-UC30, Labman Scientific Instruments, Chennai, India) at 40 kHz for 30 min with a power output of 120 W, followed by centrifugation at 10,000 rpm for 20 min. The final product was CNPs with an average size of 34 ± 5 nm. Production efficiency (83.5%) was calculated as the ratio of dried nanoparticle weight to the initial chitosan weight.

2.3. Synthesis of CNP-CTS/Starch Composite Films

The fabrication of CNP-CTS/Starch composite thin films was conducted *via* a systematic solvent-casting method. Initially, a chitosan solution was prepared by dissolving 2.00 g of CTS flakes in 100 mL of a 2% (*v/v*) aqueous acetic acid solution under constant mechanical stirring for 5–7 h until a clear chitosan acetate solution was obtained [14]. Simultaneously, 2.0 g of soluble potato starch was dispersed in 100 mL of deionized water and heated to 80°C with continuous stirring to achieve complete gelatinization and dissolution.

The composite films were formulated using a 1:1 (*w/w*) ratio of chitosan to starch. Chitosan nanoparticles (CNPs), synthesized separately, were incorporated at concentra-

tions of 2, 4, 6, and 8 wt% relative to the total CTS/Starch weight. To ensure uniform dispersion and prevent agglomeration, the CNPs were dispersed in deionized water and subjected to ultrasonication for 2 h using an ultrasonic bath (Labman Scientific Instruments, Chennai, India). These nanoparticle suspensions were then introduced dropwise into the blended CTS/Starch solution under continuous stirring for 3 h. The resulting mixtures were degassed to eliminate entrapped air bubbles, ensuring a homogeneous distribution of CNPs throughout the polymer matrix.

Following degassing, 25 mL of each composite solution was cast into borosilicate glass petri dishes (10 cm diameter, Tarsons, Tarsons Products Ltd., Kolkata, India) placed on a leveled surface. The films were vacuum-dried at 50 °C for 24 h to achieve a smooth morphology and uniform thickness. Once dried, the films were carefully detached and conditioned prior to characterization. The samples reinforced with 2, 4, 6, and 8 wt.% CNPs were coded as B, C, D, and E, respectively, while the neat CTS/Starch (1:1) film served as the control and was coded as sample A. The final film thickness ranged from 0.030 mm to 0.051 mm. For each specimen, six random measurements were recorded using a digital micrometer to ensure statistical accuracy and representativeness. The complete fabrication process is schematically illustrated in Figure 1.

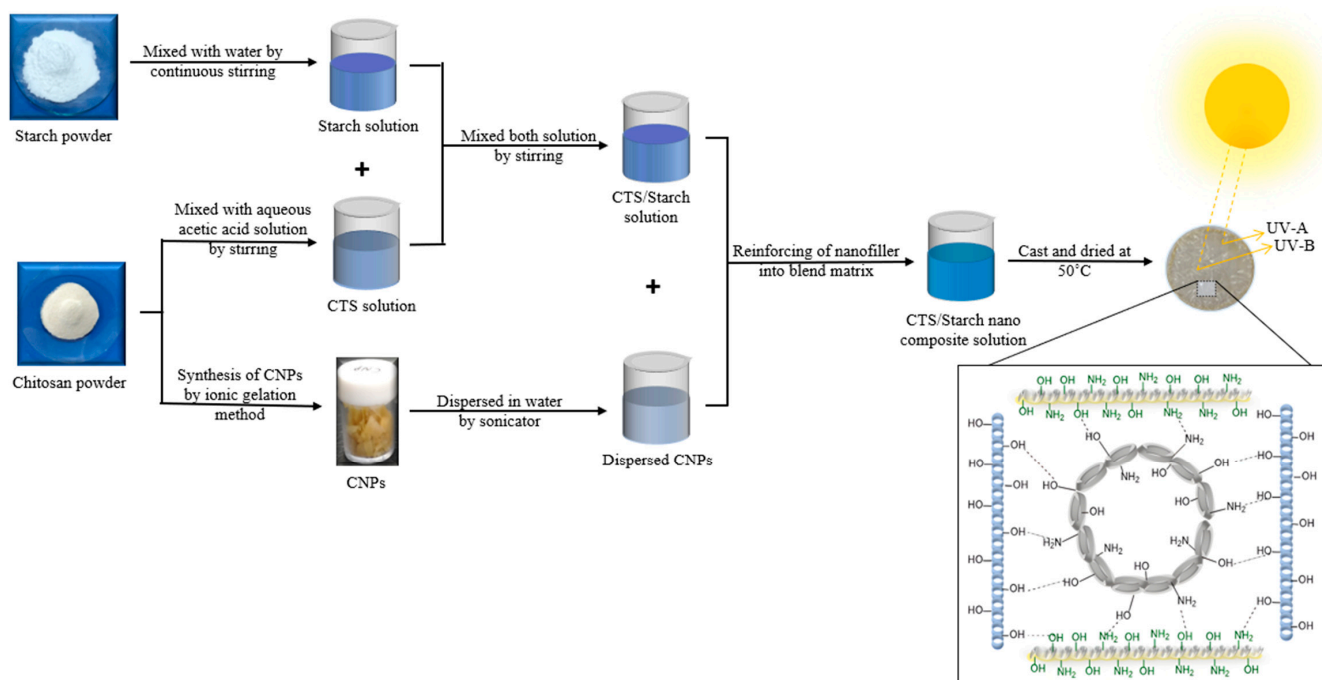


Figure 1. Schematic representation of the preparation of CTS/Starch nanocomposite films reinforced with CNPs.

2.4. Conditioning

Prior to further testing, the prepared films were conditioned at 25 ± 1 °C and $70 \pm 2\%$ relative humidity (RH) for 48 h in a desiccator. The RH was maintained using a saturated sodium chloride (NaCl) solution placed at the bottom of the desiccator, following ASTM D618-13 standard practice for conditioning plastics prior to testing [ASTM D618-13, 2013] [20]. All experiments were performed in triplicate to ensure reproducibility and to confirm the reliability of the results.

2.5. FTIR

FTIR spectra of the samples were obtained using a TENSOR 27 spectrometer (Bruker Corporation, Billerica, MA, USA). Each spectrum was recorded as the average of 32 scans

at a resolution of 4 cm^{-1} over the wavenumber range of $500\text{--}4000\text{ cm}^{-1}$. All FTIR spectra were normalized to absorbance intensity to facilitate comparison of characteristic peaks among different film formulations.

2.6. X-Ray Diffraction (XRD)

X-ray diffraction (XRD) measurements were carried out on a D2 PHASER X-ray diffractometer (Bruker Corporation, Billerica, USA) fitted with a $\text{CuK}\alpha$ radiation source ($\lambda = 0.154\text{ nm}$) to characterize the crystalline structure of the samples. The instrument operated at 40 kV and 40 mA. The scattered radiation was recorded at ambient temperature in the 2θ range of $10\text{--}40^\circ$ using a step interval of 0.02° and a scan speed of $1^\circ/\text{min}$.

2.7. Field Emission Electron Microscopy (FE-SEM)

For cross-sectional SEM imaging, the films were cryo-fractured by immersion in liquid nitrogen and snapped to obtain clean fracture surfaces. The fractured samples were mounted on aluminum stubs with conductive carbon tape and sputter-coated with a $\sim 10\text{ nm}$ gold layer using a JEOL JSM-7500F (JEOL Ltd. Akisima, Tokyo, Japan) sputter coater prior to observation.

2.8. Transmission Electron Microscopy (TEM)

Carbon film-covered copper grids ($5\text{--}6\text{ nm}$ film thickness, 200 mesh, EMS FF200 (Elioix Inc., Tokyo, Japan)) were used for imaging CNP. One drop of the CNP suspension (0.1 mg/mL) was placed on a grid for 30 min and wicked away with a filter paper. Further, the samples were stained by depositing a drop of (2 wt.%) phosphotungstic acid solution on the grid for 20 min and wicking the excess solution away with a filter paper. The grid was dried in the oven at 70°C before insertion into the microscope. Finally, JEOL TEM JEM-2100F (JEOL Ltd. Akisima, Tokyo, Japan) was utilized for the visualization and imaging of the CNPs, operating at an accelerating voltage of 200 kV.

2.9. Thermogravimetric Analysis (TGA)

Thermal stability of the films was evaluated using a thermogravimetric analyzer (TA Q50 (TA Instruments, New Castle, DE, USA)). Measurements were performed from ambient temperature to 700°C at a constant heating rate of $10^\circ\text{C min}^{-1}$, under nitrogen atmospheres. The Integral Procedure Decomposition Temperature (IPDT), as introduced by Doyle in 1961 [21] is a valuable parameter derived from the entire TGA curve. IPDT integrates the overall degradation profile into a single numerical value, offering a comprehensive assessment of a material's inherent thermal stability. In the present study, the IPDT was calculated as follows:

$$\text{IPDT } (^\circ\text{C}) = A^*K^*(T_f - T_i) + T_i \quad (1)$$

$$A^* = \frac{S_1 + S_2}{S_1 + S_2 + S_3} \quad (2)$$

$$K^* = \frac{S_1 + S_2}{S_1} \quad (3)$$

where A^* is the area ratio of the total experimental curve defined by the entire TGA thermogram. T_i is the initial experimental temperature and T_f is the final experimental temperature. Representations of S_1 , S_2 , and S_3 for calculating A^* and K^* are detailed in previous research papers [21,22].

2.10. Thickness

Film thickness, ranging from 0.030 mm to 0.051 mm, was determined using a hand-held Teclock dial thickness gauge (SM-112, TECLOCK Co., Ltd., Tokyo, Japan). For each film, six measurements were taken at random positions, and the average value was used for analysis.

2.11. Density

Film density was calculated based on the ratio of specimen weight to volume. Each specimen was weighed using an analytical balance (Wensar, Chennai, India) with a precision of 0.1 mg. Volume was determined from the measured area and thickness of the specimen. Thickness measurements were obtained using a Teclock dial thickness gauge (SM-112, TECLOCK Co., Ltd., Tokyo, Japan) with a precision of 0.01 mm, taking six readings at random positions on each specimen and using the average value for calculations. The densities of films A, B, C, D, and E are found 0.50 ± 0.01 , 0.47 ± 0.02 , 0.50 ± 0.08 , 0.33 ± 0.0081 and 0.56 ± 0.0081 , respectively, expressed as the mean of three specimens per film type. All films exhibited comparable density values, indicating that the incorporation of CNPs had no significant influence on film density—a result likely attributable to the low loading level of CNPs.

2.12. Optical and UV Visibility

The opacity and transmittance of CTS/Starch nanocomposite films were measured using a JASCO V-650 UV–Vis spectrophotometer (JASCO Corporation, Tokyo, Japan) in transmission mode. Rectangular film specimens ($2 \times 2 \text{ cm}^2$) were placed directly on the instrument's sample holder window. Absorbance was recorded at 600 nm, and opacity (mm^{-1}) was calculated using Equation (4) [23]:

$$\text{Opacity} = \frac{\text{Abs}_{600}}{d} \quad (4)$$

where d is the film thickness (mm). Transmittance values were also obtained across the UV–Vis range. All measurements were performed in triplicate, and average values are reported.

To determine the blocking effect (B_{eff}) of CNP, we applied a method from a previous study to the CTS/Starch-based films [23].

$$B_{\text{eff}} = \frac{T_{\text{CTS/Starch}} - T_{\text{CNP-CTS/Starch}}}{\text{Loading of CNP}} \quad (5)$$

Here, $T_{\text{CTS/Starch}}$ and $T_{\text{CNP-CTS/Starch}}$ denote the transmittance values of the pure CTS/Starch film and the CNP-enhanced CTS/Starch nanocomposite, respectively.

2.13. Water Absorbency (WA) of the Films

The WA capacity of the prepared films was assessed by determining their percentage swelling ratio. The experimental procedure and test conditions followed those outlined in our recently published work [23]. Samples were immersed in water and remained undisturbed for 24 h to reach swelling equilibrium.

$$\text{Swelling Ratio}(\%) = P_s = \frac{W_s - W_d}{W_d} \times 100 \quad (6)$$

where W_d and W_s denote the weights of the dry and swollen film samples, respectively.

2.14. Equilibrium Moisture Content (EMC)

The equilibrium moisture content (EMC) of the films was assessed using square specimens measuring $2 \times 2 \text{ cm}^2$. Each sample was oven dried at $102 \pm 2 \text{ }^\circ\text{C}$ until a constant mass (W_∞) was achieved, indicating moisture equilibrium. This final mass was compared with the initial dry mass (W_0), and EMC was calculated according to [14]:

$$\text{EMC} = \frac{W_0 - W_\infty}{W_0} \times 100 \quad (7)$$

2.15. Water Vapor Permeation (WVP)

The water vapor permeability (WVP) of CTS/Starch nanocomposite films was determined following the ASTM E96/E96 05 standard protocol, as described by Yadav et al. [15]. For each test, 20 mL of distilled water was placed in a petri dish, ensuring a gap of approximately 1 cm between the water surface and the mounted film. The films were affixed to the dish openings using a water-resistant sealant to prevent leakage. The sealed dishes were then conditioned in a humid chamber at $27 \text{ }^\circ\text{C}$ to maintain 75% relative humidity (RH), with their weights recorded at predetermined time intervals. WVTR values ($\text{g}\cdot\text{m}/(\text{m}^2\cdot\text{s}\cdot\text{Pa})$) obtained from the gravimetric cup method were used to calculate WVP according to Equation (8) [14]:

$$\text{WVP} (\text{g}\cdot\text{m}/(\text{m}^2\cdot\text{s}\cdot\text{Pa})) = \frac{\text{WVTR} \cdot L}{\Delta P} \quad (8)$$

where L is the film thickness (m) and Δp is the vapor pressure difference. The vapor pressure difference (Δp) was calculated as the difference between the saturated vapor pressure of water at $27 \text{ }^\circ\text{C}$ (3.57 kPa) and the partial vapor pressure of water in the chamber atmosphere (2.68 kPa, corresponding to 75% RH). Thus, Δp was taken as 0.89 kPa in all calculations of WVP.

2.16. Mechanical Properties

The mechanical properties of the films were assessed in accordance with ASTM D638 [17] standards using a Tech plast model UTM-192-2L (Techplast testing Machines, Ahmedabad, India) equipped with a 500 N load cell, operated at $24 \pm 2 \text{ }^\circ\text{C}$. All tensile tests were performed at a constant crosshead speed of 10 mm min^{-1} . Film specimens were prepared in rectangular form, measuring $10 \times 60 \times 0.04 \text{ mm}$, prior to testing.

2.17. Biodegradability Test

Soil burial experiments to evaluate the biodegradation behavior of the films were performed following the protocol outlined in earlier reports [14,24]. Film specimens were prepared in rectangular dimensions ($1 \text{ cm} \times 1 \text{ cm} \times 0.004 \text{ cm}$) and oven-dried at $100 \text{ }^\circ\text{C}$ for 5 h to obtain their initial weight (w_i). A 30 g portion of soil was placed in a plastic pot, forming a layer approximately 1 cm thick, into which the samples were embedded at a depth of 0.5 cm below the surface. The test was conducted under controlled conditions of $25 \text{ }^\circ\text{C}$ and 30% relative humidity. At predetermined intervals, the buried films were carefully retrieved, gently cleaned with tissue paper to remove adhering soil particles, oven-dried at $105 \text{ }^\circ\text{C}$ for 6 h, and reweighed (w_t). The extent of biodegradation was expressed as percentage weight loss, calculated using Equation (9) [25]:

$$\text{Weight loss (\%)} = \frac{(W_i - W_t)}{W_i} \times 100 \quad (9)$$

3. Results

3.1. FTIR

Polysaccharide infrared spectroscopy has been conducted in numerous studies. Due to its quick, strong, and non-destructive nature, the FTIR technique is frequently employed to examine the miscibility of renewable polymers and nanomaterials. Variations in FTIR spectra—such as shifts in characteristic absorption bands—serve as indicators of interactions between chemical functional groups. Such spectral modifications often suggest enhanced compatibility and good miscibility within the polymer system [23].

The distinctive absorption bands were visible at 1653 cm^{-1} in the CTS spectrum (Figure 2, assigned to the amide I ($\text{C}=\text{O}$ stretching)), 1550 cm^{-1} assigned to the amide II (N-H bending modes), the broad band at 3515 cm^{-1} was the OH stretching and the peak at 1418 cm^{-1} was assigned to $-\text{C-O}$ stretching of the primary alcoholic group in CTS. The intermolecular interaction of CNPs is revealed by FTIR analysis. The peak shifted to 1682 cm^{-1} and 1578 cm^{-1} for CNP due to the interaction between the phosphoric group of TPP and the amino group of CTS in nanoparticles [26]. The OH peak shifted from 3515 cm^{-1} to 3557 cm^{-1} in the CNP spectrum. In the starch film spectrum, the broad absorption band at 3466 cm^{-1} corresponds to O-H stretching vibrations, whereas the peaks at 1528 cm^{-1} and 1250 cm^{-1} are attributed to $\delta(\text{O-H})$ bending of bound water and CH_2 scissoring vibrations, respectively [27]. Variations in the distinctive spectrum peaks of two components mixed together influence the physical blends vs. chemical interactions [28]. As starch was added, the amino peak of the CTS in the typical CTS/Starch composite film spectra moved from 1550 cm^{-1} to 1483 cm^{-1} . These results indicate the presence of interactions between the hydroxyl groups of starch and the amino groups of chitosan [28,29]. Upon incorporation of CNPs, the absorption band at 1483 cm^{-1} shifted to 1493 cm^{-1} (broader to sharper peak) and at 3472 cm^{-1} shifted to 3538 cm^{-1} (sharper to broader), confirming the formation of hydrogen bonding interactions between the CTS/Starch matrix and the CNPs [30].

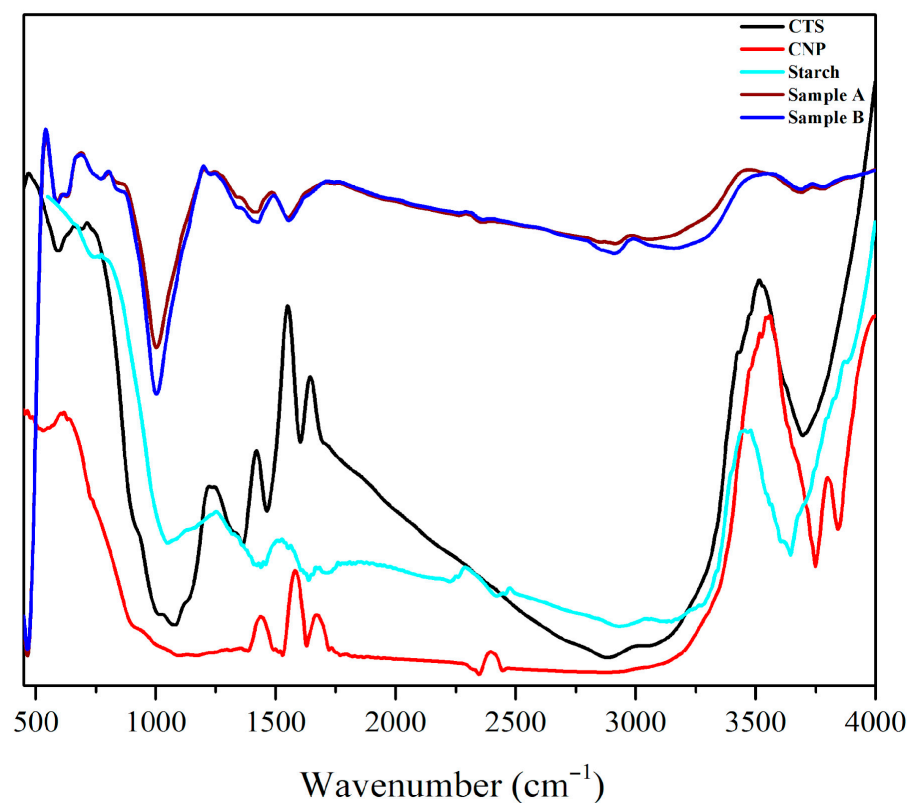


Figure 2. FTIR spectra of CTS, CNP, Starch, Sample A and Sample B.

3.2. XRD

The crystalline characteristics of the prepared nanocomposite films were investigated using X-ray diffractometry, providing insight into the miscibility and compatibility of the film's various components. This analysis helped to determine any physical or chemical interactions between the nanoparticles and the biopolymer matrix that could influence the overall film properties. Figure 3a,b present the XRD patterns of CTS, CNP, starch, and the CNP-incorporated CTS/Starch composites (samples B, C, D, and E).

Although CTS is generally considered semi-crystalline to amorphous, the sharper peaks observed in our XRD pattern may be attributed to residual crystallinity inherited from chitin, the degree of deacetylation, and the specific film preparation conditions that promote molecular ordering [31]. Similar observation has been reported in the literature under controlled processing conditions, suggesting that the crystalline domains observed here are not unusual but rather preparation-dependent [32].

The X-ray diffraction pattern of chitosan powder displayed two major diffraction peaks at $2\theta = 11.59^\circ$ and 20.20° , confirming its crystalline state, consistent with previous reports [27–29]. In the case of CNP, these characteristic CTS peaks weakened, while new peaks emerged at $2\theta = 11.98^\circ$, 18.42° , and 24.24° , which are in full agreement with published data [33,34]. The CTS/Starch matrix, however, showed a broad diffraction peak at $2\theta = 20.33^\circ$, indicative of its low crystalline structure, similar to earlier findings [35,36].

The XRD profile of starch films exhibited characteristic peaks at 15° , 17° , 18° , and 23° (2θ), confirming their semi-crystalline nature. In the CTS/Starch nanocomposite films, these peaks were present but with reduced intensity, suggesting partial disruption of starch crystallinity due to chitosan incorporation and nanoparticle reinforcement. This indicates improved molecular interactions and compatibility within the polymer matrix.

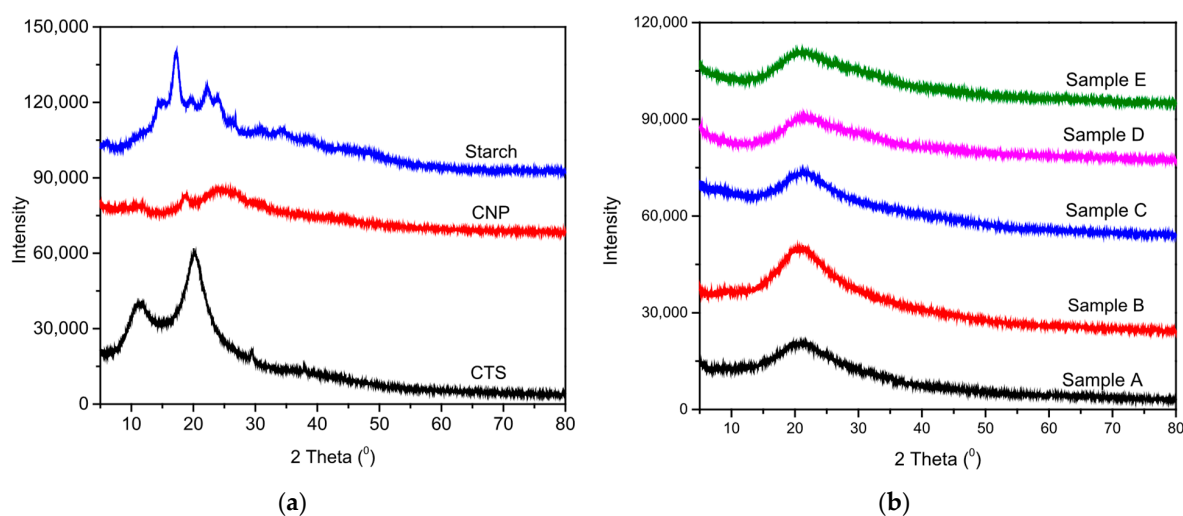


Figure 3. XRD of (a) CTS, CNP and Starch, (b) Sample A, Sample B, Sample C, Sample D and Sample E.

Significantly, Figure 3b showed that the inclusion of CNPs into the CTS/Starch matrix did not alter the position of its diffraction peaks. Furthermore, no new peaks corresponding to CNPs (i.e., peaks at $2\theta = 11.98^\circ$, 18.42° , and 24.24°) were observed in the nanocomposite films [37,38]. These findings collectively suggest that the addition of CNPs at different weight percentages did not disrupt the polymer's amorphous structure, but rather indicated that the CNPs were successfully incorporated between the polymer chains. This effect can be attributed to the homogeneous dispersion and uniform distribution of CNPs within the polymer matrix, which preserved the amorphous structure of the CTS/Starch system throughout the film-forming process.

3.3. SEM

Scanning electron microscopy (SEM) analysis offered detailed visualization of the composite matrix, revealing the morphology, particle size, spatial distribution, and interfacial interactions of the embedded nanoparticles. The surface morphology of the CTS/Starch blend and CNPs-incorporated films is displayed in Figure 4. A highly insightful comparison between the surface of the blend (Sample A, Figure 4a) and the CNP-loaded composite (Sample D, Figure 4c) revealed a remarkably smooth and uniform surface, free from visible large aggregates or phase separation. This homogeneity strongly indicated excellent miscibility and compatibility between the CNPs and the CTS/Starch polymer matrix. Such uniform distribution is paramount for the nanoparticles to effectively contribute their reinforcing and barrier-enhancing effects throughout the film [39]. Cross-sectional SEM images (Figure 4b,d) provided strong evidence of effective CNP integration. Figure 4b shows the control film's dense and homogeneous internal structure, serving as a vital reference. Notably, the CNP-loaded composite film (Figure 4d) maintained this compact structure without large voids or cracks, indicating that CNPs were well-embedded within the polymer network. This deep embedding and the resulting extensive interfacial contact between CNPs and polymer chains are critical for efficient stress transfer, enhancing mechanical strength, and for creating a tortuous path that improves barrier properties against permeating molecules. From the SEM images of CNPs (Figure 4e), TEM observations (Figure 4f), and the enhanced film properties collectively validate the successful formation and stability of CNPs.

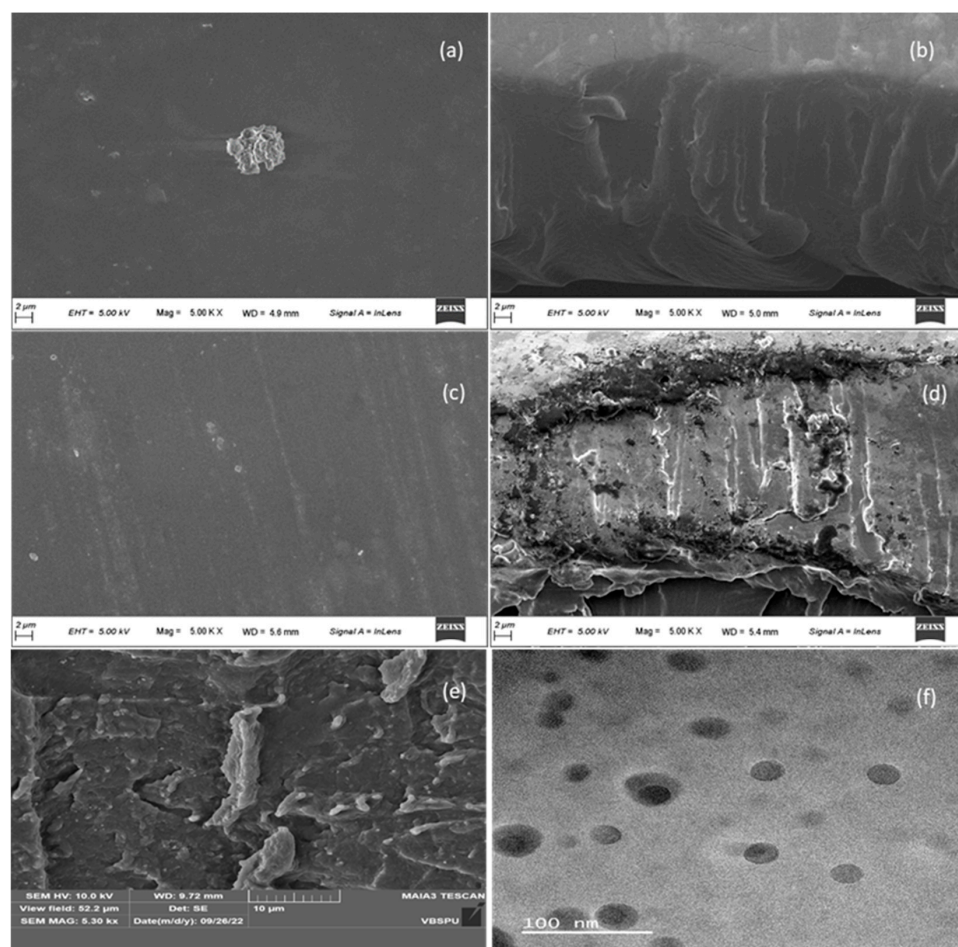


Figure 4. FESEM of (a) surface image of Sample A, (b) cross-section image of Sample B, (c) surface image of Sample D, (d) cross-section image of Sample D, (e) image of CNP powder and (f) TEM image of CNP.

3.4. TEM of Chitosan Nanoparticles (CNPs)

TEM is a strong method for high-resolution visualization and analysis of a material's microstructure. Figure 4f clearly depicts well-defined, mostly spherical nanoparticles that appear to be well-dispersed, with minimal significant aggregation within the observed field. The average particle size was determined from TEM micrographs by measuring at least 100 nanoparticles using Image J software, yielding an average size of 34 ± 5 nm [40]. This TEM image provides definitive proof of the nanoparticles' nanoscale dimensions and uniform morphology, which are critical for their effectiveness as reinforcing agents.

3.5. Thermogravimetric Analysis (TGA)

The TGA technique is very useful for studying unique decomposition patterns of all types of polymers. It provides information about the polymer's stability and thermal degradation. TGA measures a polymer's weight loss as a function of temperature, providing insights into its thermal degradation behavior. Figure 5 illustrates the thermogravimetric analysis (TGA) curves for samples A and B. For sample A, an initial weight loss of approximately 15 wt% around 100 °C (commencing at ~50 °C) was attributed to the evaporation of absorbed water. Its polymer decomposition temperature (PDT) was found to be approximately 140 °C. The rate of weight loss increased significantly between 175 °C and 245 °C before decreasing. A maximum weight loss of about 82 wt% was recorded around 700 °C. The integral decomposition temperature (IPDT) (Table 1) and final decomposition temperature (FDT) for sample A were determined to be 573 °C and 670 °C, respectively. Similarly, for sample B, an initial 14 wt% weight loss at approximately 100 °C is attributed to absorbed water. Degradation of sample B (2 wt% loading) began around 150 °C, with its PDT identified at 170 °C. The weight loss rate accelerated from 210 °C to 350 °C before gradually declining. A maximum weight loss of 78 wt% was observed near 700 °C. Sample B's IPDT and FDT were 553 °C and 650 °C, respectively. A direct comparison of the thermograms revealed that sample A exhibited higher FDT and IPDT values (Table 1) than sample B, indicating that sample A possesses superior thermal stability.

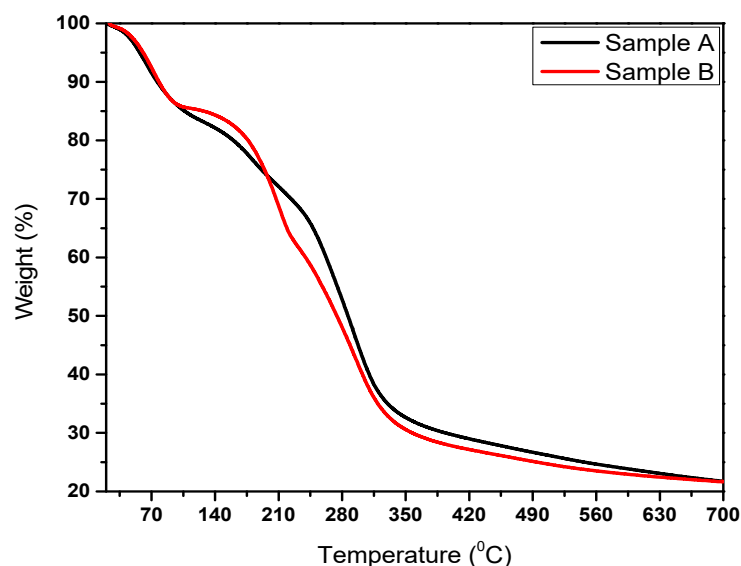


Figure 5. TGA Curve of Samples A and B.

Table 1. Thermal behavior of samples A and B.

Samples	T _i (°C)	T _f (°C)	S ₁	S ₂	S ₃	A*	K*	IPDT (°C)
A	22	594	16,449	13,664	27,087	0.53	1.83	573
B	24	596	16,055	13,112	28,036	0.51	1.82	553

3.6. Water Absorbency (WA)

Table 2 displayed the WA of the nanocomposite films. The WA of the CTS/Starch films significantly reduced in the presence of CNPs. As an example, the weight average of the sample A film was $340 \pm 1.63\%$; however, this dropped to $244.48 \pm 1.13\%$ after 2 wt.% of CNP were loaded into the film. When the CNP loading reached 8 wt.%, the WA values dropped to $88.65 \pm 1.12\%$. This behavior could be explained by the film's higher CNP content, which lowered the amount of free hydroxyl groups in the polymeric chain and reduced the hydrogen bonding contact that water molecules had with the CNP-CTS/Starch nanocomposite [14,41]. A similar finding was reported by Arredondo et al. [41] in gellan gum films reinforced with eggshell nanoparticles.

Table 2. Values of thickness, WA, EMC, WVP, opacity, transmittance, TS, and EB of samples A, B, C, D and E.

Sample Name	(0 wt.%) CNP-(CTS: Starch) (1:1)	(2 wt.%) CNP-(CTS: Starch) (1:1)	(4 wt.%) CNP-(CTS: Starch) (1:1)	(6 wt.%) CNP-(CTS: Starch) (1:1)	(8 wt.%) CNP-(CTS: Starch) (1:1)
Sample code	A	B	C	D	E
Thickness (mm)	0.051 ± 0.001	0.030 ± 0.0081	0.036 ± 0.0008	0.035 ± 0.0008	0.047 ± 0.0008
WA (%)	340.00 ± 1.63	244.48 ± 1.13	142.59 ± 1.01	112.41 ± 0.87	88.65 ± 1.12
EMC (%)	16.52 ± 1.03	15.41 ± 0.88	14.62 ± 0.94	13.30 ± 0.87	12.50 ± 1.05
WVP ($\times 10^{-11}$ $\text{g}\cdot\text{m}/(\text{m}^2\cdot\text{s}\cdot\text{Pa})$)	6.18 ± 0.54	5.54 ± 1.14	5.44 ± 1.01	5.28 ± 0.90	5.38 ± 0.93
Opacity (mm^{-1})	2.52 ± 1.02	2.38 ± 1.01	3.51 ± 1.08	4.83 ± 0.83	4.68 ± 0.93
Transmittance (%)	89.9 ± 0.80	89.20 ± 0.5	84.20 ± 0.7	81.30 ± 0.8	79.70 ± 1.2
TS (MPa)	6.38 ± 1.16	11.48 ± 0.96	15.43 ± 0.91	16.49 ± 0.93	15.38 ± 0.85
EB (%)	14.42 ± 0.72	61.69 ± 0.94	70.73 ± 0.94	98.78 ± 0.83	69.42 ± 0.76

3.7. Equilibrium Moisture Content (EMC)

Water sensitivity and hygroscopic qualities determine a biopolymer's food packaging film's capacity to absorb moisture. The EMC values for films A, B, C, D, and E, as presented in Table 2, were $16.52 \pm 1.03\%$, $15.41 \pm 0.88\%$, $14.62 \pm 0.94\%$, $13.3 \pm 0.87\%$ and $12.5 \pm 1.05\%$, respectively. The moisture content decreased somewhat after the CNPs were included. The EMC results are higher in sample A than in the other films. This could be as a result of interactions in the composite formulation that increased susceptibility to water and improved the affinity of the material to bind water molecules because of exposure to the numerous hydroxyl groups included in both structures [42].

3.8. Water Vapor Permeation (WVP)

The WVP of CTS/Starch and CNP-CTS/Starch nanocomposite films are shown in Table 2. Since the primary purpose of the packing films is to evaluate their moisture permeability, Water Vapor Permeability (WVP) is the main parameter that was examined. The WVP of sample A film was $6.18 \pm 0.54 \text{ g}\cdot\text{m}/(\text{m}^2\cdot\text{s}\cdot\text{Pa})$ that decreased significantly to $5.38 \pm 0.93 \text{ g}\cdot\text{m}/(\text{m}^2\cdot\text{s}\cdot\text{Pa})$ (Sample E). It decreased remarkably 12.94% compared to the sample A film. This effect is primarily caused by the strong affinity between the CTS/Starch and CNP. This interaction tightens the film's matrix and reduces its internal porosity, thereby limiting the passage of light. Therefore, the result indicated that the low WVP values of CNPs incorporated in CTS/Starch nanocomposite films (samples B, C, D and E) would be more suitable for packaging applications [42].

3.9. Opacity and UV Visibility

The optical characteristics of the transparent food packaging films were assessed by investigating their opacity. Opacity defines the degree to which a substance prevents light

from passing through it. While transparent films are generally preferred, opaque films are recommended when light exposure leads to food deterioration [43]. Figure 6 visually represents the opacity of the films, including a digital image of the CTS/Starch films, and Table 2 presents the corresponding transparency and opacity values.

From Table 2, it is evident that sample A, the control film, displayed an opacity of $2.52 \pm 1.02 \text{ mm}^{-1}$, which was significantly lower than films incorporated with CNPs. All composites (samples B, C, D, E) clearly exhibited higher opacity values compared to sample A, directly attributable to the increased CNP loading. This indicates that the composites effectively prevent light transmission. Specifically, opacity increased from $2.52 \pm 1.02 \text{ mm}^{-1}$ to $4.68 \pm 0.93 \text{ mm}^{-1}$ as the CNP loading rose from 0 to 8 wt%, as detailed in Table 2. Opacity was calculated using the absorbance at 600 nm.

Figure 7a illustrates the UV absorbance graphs for the CTS/Starch film and its CNP composites. UV light absorption increased with higher CNP content, likely due to the presence of CNP's three-dimensional structure. Furthermore, CNPs effectively blocked UV light, rendering the films more resistant to UV wavelengths. This aligns with findings by Zhang et al. [44], who reported reduced UV absorption in Cellulose nanocrystal reinforced PVC thin films.

All films showed decreased transmittance (Table 2) values when exposed to UV light (200–380 nm) compared to visible light (380–800 nm). As depicted in Figure 7b, the transmittance of CNP-containing films steadily dropped relative to the neat blend film, confirming CNP's UV-blocking capability. Figure 7c shows the variation in transmittance with CNP loading at 800 nm. At 8 wt.% CNP content, the CTS/Starch film's transmittance decreased from $89.9 \pm 0.80\%$ to $79.70 \pm 1.2\%$. Overall, while CNP addition enhanced the UV light resistance of the bio-nanocomposite films, it simultaneously decreased their transparency. The UV characteristics of these CNP-CTS/Starch nanocomposite films are consistent with previously published works [14,17]. The UV-blocking efficiency (B_{eff}) of the CNPs was evaluated at key wavelengths—300 nm (UV-B) and 500, 700, and 750 nm within the visible spectrum—as illustrated in Figure 7d. Notably, films containing 6 wt.% CNPs demonstrated the highest B_{eff} across the entire UV-Visible range, indicating superior UV absorption capability at this concentration. These findings affirm that integrating CNPs into CTS/Starch matrices markedly improves their barrier performance, particularly against UV radiation, making them highly suitable for light-sensitive packaging applications.



Figure 6. Digital images illustrating the transparency and opacity of the prepared films. (a) A representative thin film was placed on a petri dish without any background visualization. (b) CTS/Starch film (sample A). (c–f) CNP-CTS/Starch nanocomposite films with progressively increasing CNP loading (Samples B, C, D, and E, respectively). Images (b–f) are positioned over the logo of Veer Bahadur Singh Purvanchal University to visually demonstrate the films' decreasing transparency and increasing opacity with higher CNP content.

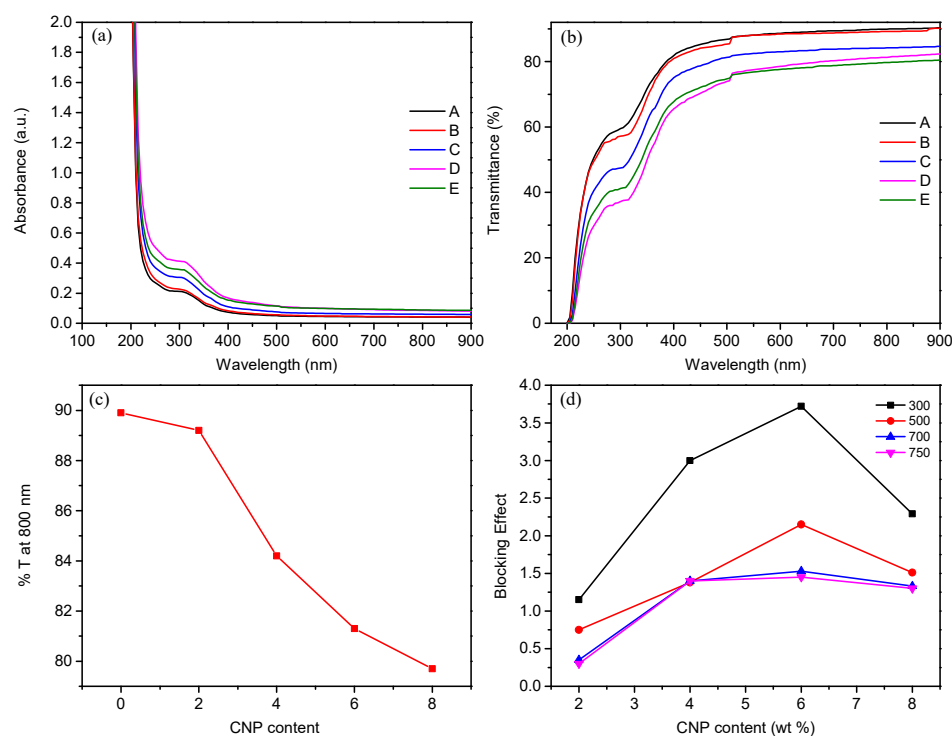


Figure 7. (a) UV absorption curve, (b) UV transmittance curve, (c) Percentage transmittance (%T) of UV at 800 nm (d) B_{eff} of UV at different wavelengths for samples B, C, D, E.

3.10. Mechanical Properties

The mechanical resilience of packaging films plays a pivotal role in preserving the structural integrity of food products throughout their lifecycle. These properties are critical in ensuring both the safety and quality of packaged goods. Two key indicators—tensile strength (TS) and elongation at break (EB)—serve as benchmarks for assessing film performance. TS reflects the maximum tensile load a film can withstand before rupture, offering resistance against tearing or puncturing during handling, transport, and storage. EB, on the other hand, measures the film's stretchability prior to failure, highlighting its flexibility and capacity to accommodate the expansion or deformation of food items without compromising the package [45].

Robust mechanical characteristics are essential for edible films intended for food industry applications, as they ensure structural integrity throughout processing, handling, and transportation [46]. In this context, the mechanical behavior of CTS/Starch blend films infused with varying concentrations of CNPs was systematically assessed using standard tensile testing. Key performance metrics—tensile strength (TS), indicating the film's resistance to rupture under tension, and elongation at break (EB), reflecting its flexibility and stretchability—were used to quantify the impact of CNP incorporation on film durability.

To evaluate the mechanical performance of the composite films, TS and EB were calculated as the average of five independent measurements for each formulation. As detailed in Table 2, even minimal incorporation of CNPs led to a substantial enhancement in tensile properties. Specifically, the TS values for samples B (11.48 ± 0.96 MPa), C (15.43 ± 0.91 MPa), D (16.49 ± 0.93 MPa), and E (15.38 ± 0.85 MPa) significantly surpassed that of the control sample A (6.38 ± 1.16 MPa), reflecting increases of 79.93%, 141.85%, 158.40%, and 141.10%, respectively. This improvement is likely driven by strong hydrogen bonding interactions between CNPs and the CTS/Starch matrix, which reinforce the polymer network [28,47,48]. However, when the CNP content was raised from 6 wt.% to 8 wt.%, a decline in TS from 16.50 MPa to 15.08 MPa was observed, possibly due to nanoparticle ag-

glomeration disrupting uniform stress distribution within the matrix [49,50]. Similar trends have been reported in the literature. Tabbasum et al. noted a decrease in TS from 24.42 MPa to 20.13 MPa [51] upon loading 5 wt.% ZnO nanoparticles into a chitosan–xanthan gum blend. Other studies have explored the mechanical impact of low nanofiller concentrations in commercial biopolymers [52,53]. For instance, silver nanoparticle incorporation into chitosan–starch films yielded TS enhancements of 69.60%, 68.95%, and 74.56% over a baseline of 66.81% [54]. Additionally, mechanical testing of starch–polyvinyl alcohol films loaded with 5% CNPs revealed a TS increase from 35 MPa to 47 MPa, accompanied by a 40% reduction in EB—suggesting that higher nanoparticle content may compromise flexibility despite strengthening the film [16].

The TS of the CTS/Starch blend film measured in this study (6.38 ± 1.16 MPa) falls within the spectrum of values reported in earlier research. Comparable TS values include approximately 37.5 MPa [48] 35 MPa [28], 40 MPa [27], 5 MPa [12], 9.9 MPa [55], 9.33 MPa [56] and 66.81 MPa [54]. This variation underscores the influence of formulation strategies, processing conditions, and nanoparticle incorporation on the mechanical performance of biopolymer-based films.

The TS values observed for the developed films exhibited a performance range comparable to conventional plastic-based packaging materials. For reference, TS values for commonly used polymers include Low-density polyethylene (LDPE, 8–10 MPa), high-density polyethylene (HDPE, 19–31 MPa), ethylene vinyl alcohol (EVOH, 6–19 MPa), polycaprolactone (PCL, 4 MPa), polystyrene (PS, 31–49 MPa), polylactic acid (PLA, 45 MPa), polyvinyl chloride (PVC, 42–55 MPa), and polypropylene (PP, 27–98 MPa) [57]. In a related study by Wardana et al. [58], the TS of composite films showed a progressive increase from 57.82 MPa to 60.46 MPa and peaked at 80.67 MPa as the chitosan nanoparticle content was raised from 0 to 0.1 and 0.5 wt%. Interestingly, further increasing the chitosan nanoparticle concentration to 1 wt.% led to a decline in TS to 46.76 MPa, likely due to nanoparticle agglomeration disrupting the polymer matrix uniformity.

Shapi'I et al. [59] also documented that the incorporation of chitosan nanoparticles significantly enhanced the tensile strength of starch-based biopolymer films. This observation aligns with the findings of Moura et al. [15], who investigated the impact of CNP loading on the mechanical behavior of carboxymethylcellulose films. Their study revealed a notable increase in tensile strength—from 5 MPa to 32 MPa—as the CNP concentration rose from 0% to 40% *w/w*, highlighting the reinforcing potential of nanoparticles in biopolymer matrices.

Conversely, the elongation at break (EB%) values for samples A through E were recorded as 14.42 ± 0.72 , 61.69 ± 0.94 , 70.73 ± 0.94 , 98.78 ± 0.83 , and 69.42 ± 0.76 , respectively (Table 2). These results reveal a pronounced enhancement in film flexibility with increasing CNP content up to 6 wt.%, where EB rose from 327% to a peak of 584%.

However, a further increase in CNP loading to 8 wt.% led to a decline in EB to 69.20%, suggesting a threshold beyond which excessive nanoparticle concentration may hinder polymer chain mobility. This trend is likely attributed to the synergistic effect of CNPs, driven by their high aspect ratio and capacity to establish interconnected networks and hydrogen bonding interactions with chitosan molecules [60].

A comparable trend was recently reported by Wardana et al. [58], reinforcing the observation that nanocomposite films exhibit enhanced elongation at break (EB). The EB values of the developed CNP-reinforced films align well with those of conventional plastic materials, such as polystyrene (PS: 2–3%), polyvinyl chloride (PVC: 20–180%), and polyvinylidene chloride (PVDC: 10–40%). Considering the simultaneous improvements in tensile strength (TS) and EB, the CNP-integrated biopolymer films demonstrate strong potential for application in the packaging sector, offering a sustainable alternative to petroleum-based plastics.

3.11. Biodegradability Test

To combat the significant packaging waste generated by the food industry and its associated environmental problems, the adoption of biodegradable food packaging is a key requirement. In response, researchers are actively exploring sustainable biomaterials that can serve as viable alternatives to conventional plastics. Biodegradable materials are defined as those that can be broken down by the enzymatic action of living organisms [61,62]—a property well-studied in food materials [63]—offer a recyclable and environmentally sound solution compared to traditional non-biodegradable packaging.

In this context, a comparative assessment of the biodegradability of CTS/Starch films and their CNP-reinforced counterparts was undertaken to evaluate the influence of nanoparticle incorporation on environmental degradation behavior. Biodegradability in the tested films was determined by monitoring changes in surface morphology and percent weight loss over time [51].

The results, presented in Figure 8, revealed that the percent weight loss of all fabricated films (samples A–E) increased progressively with the number of burial days. Notably, sample E (8 wt% CNP loading) exhibited the highest degradation, with 14% weight loss at day 5 and 55.18% at day 15. This trend indicates that the incorporation of CNPs significantly accelerates the biodegradation process compared to the neat CTS/Starch films.

The enhanced biodegradability of CNP-reinforced films can be explained by several factors:

- Enhanced hydrophilicity and surface area: The nanoscale dimensions of CNPs increase surface roughness and hydrophilic character, facilitating greater water uptake and microbial colonization [64].
- Accelerated microbial accessibility: Chitosan itself is biodegradable, and the presence of nanoparticles provides additional sites for enzymatic attack. The uniform dispersion of CNPs within the polymer matrix (SEM/TEM, Figure 4) creates micro-domains that are more susceptible to microbial degradation [65].
- Intermolecular bonding and film disruption: Although CNPs form hydrogen bonds with starch and chitosan (FTIR, Section 3.1), these interactions introduce structural heterogeneity. Under soil burial conditions, this heterogeneity promotes faster fragmentation and weight loss compared to neat CTS/Starch films [66].

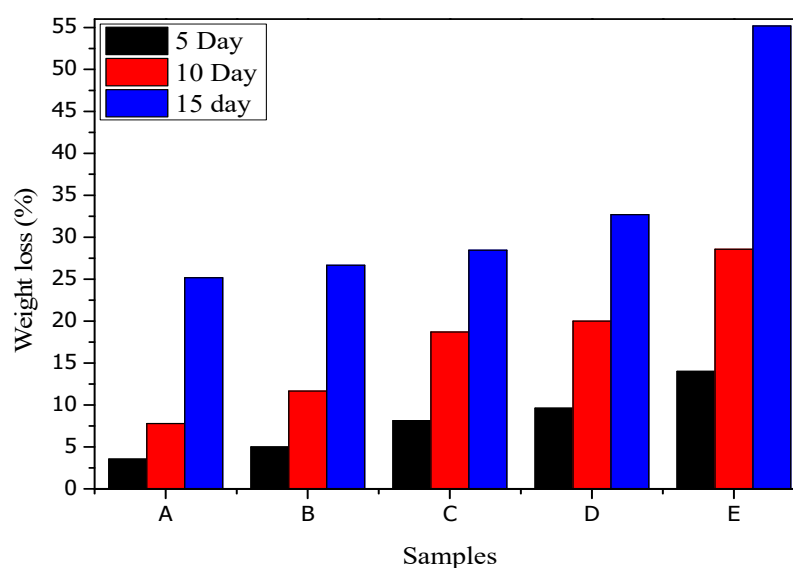


Figure 8. Biodegradability of samples A, B, C, D and E.

Thus, all fabricated films tested in the current study exhibited measurable weight loss, confirming their biodegradability [67]. Importantly, the incorporation of CNPs into CTS/Starch films not only reinforced the polymer matrix through intermolecular bonding [51] but also enhanced their environmental responsiveness, leading to faster degradation. This dual effect underscores the potential of CNP-reinforced biopolymer films as sustainable packaging materials that balance durability during use with biodegradability after disposal.

4. Conclusions

Composite blend films composed of chitosan (CTS), starch, and CNPs were synthesized via a solution mixing–evaporation technique. FTIR analysis confirmed that the incorporation of CNPs enhanced miscibility between CTS and starch through hydrogen bonding interactions. XRD results indicated uniform dispersion of CNPs within the CTS/Starch polymer matrix. The addition of starch moderately improved tensile strength, while further inclusion of CNPs significantly boosted mechanical performance. Although water barrier properties slightly declined in the CNP-CTS/Starch films compared to CTS/Starch alone, the films exhibited excellent UV shielding capabilities. Biodegradability tests affirmed the environmental sustainability of the material, with CNP-loaded composites showing reduced degradation due to stronger hydrogen bonding. Given these properties—mechanical robustness, UV protection, and biodegradability—this film is particularly suitable for packaging fresh produce (e.g., fruits and vegetables), dry snacks (e.g., chips, crackers), bakery items (e.g., bread, cookies), and minimally processed foods. Its ability to shield against UV light and maintain structural integrity makes it a promising green alternative to petroleum-based synthetic plastics in food packaging applications.

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