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ILMIY JURNALI
Vol 2 № 6 2026

RADIATION-RESISTANT COMPOSITE FILMS BASED ON WHEAT STRAW CELLULOSE

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ABSTRACT. The utilization of agricultural residues as renewable sources for advanced functional materials has attracted considerable attention in recent years, particularly in the development of environmentally sustainable radiation-shielding systems. In the present work, cellulose was isolated from wheat straw through alkaline delignification and subsequent hydrogen peroxide bleaching. The obtained cellulose was further functionalized using TEMPO-mediated oxidation and incorporated into a polyvinyl alcohol (PVA) matrix to fabricate biodegradable composite films with radiation-protective properties.

A comprehensive characterization of the extracted cellulose and the resulting composites was carried out using FTIR, XRD, SEM, TGA, tensile strength measurements, UV–Vis spectroscopy, and gamma-ray attenuation analysis. The isolated cellulose exhibited a crystallinity index of 72.4% and showed thermal degradation at approximately 348°C, confirming its potential as an effective reinforcing component. The introduction of TEMPO-oxidized cellulose into the PVA matrix resulted in notable improvements in both mechanical and thermal performance. In particular, tensile strength increased from 39.2 MPa for neat PVA films to 74.6 MPa for the cellulose-reinforced composites.

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The radiation attenuation study revealed that the developed composites provided better gamma-ray shielding performance than pure PVA, suggesting that the modified cellulose contributes not only to structural reinforcement but also to improved protective functionality. These findings demonstrate that wheat-straw-derived cellulose can serve as a valuable bio-based reinforcement for the fabrication of multifunctional composite films. The proposed materials offer a combination of biodegradability, mechanical robustness, thermal stability, and radiation-shielding capability, which may support their use in medical packaging, protective surface coatings, and other sustainable radiation-protection applications.

KEYWORDS: wheat straw cellulose; TEMPO-mediated oxidation; polyvinyl alcohol (PVA); biopolymer composites; gamma-ray attenuation; radiation-shielding materials; biodegradable films; sustainable materials.

INTRODUCTION. The use of ionizing radiation has expanded considerably over the past decades owing to its importance in nuclear energy systems, medical imaging and radiotherapy, aerospace engineering, and industrial processing. This expansion has increased the need for materials capable of maintaining their structural integrity and functional performance under radiation exposure. Conventional shielding materials, including lead-based composites, metallic barriers, and synthetic polymer systems, provide effective protection against radiation. Nevertheless, their widespread application is often limited by high weight, environmental concerns, poor degradability, and difficulties associated with end-of-life management. These limitations have encouraged researchers to investigate more sustainable alternatives based on renewable resources.

Lignocellulosic biomass has become an important source of raw materials for the development of environmentally benign functional products. Among various biomass resources, agricultural residues offer several advantages, including low cost, wide availability, and renewability. Wheat straw is particularly attractive because it is generated in large quantities worldwide and remains underutilized in many regions. Its chemical

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composition, typically consisting of 35–45% cellulose, 20–30% hemicellulose, and 15–25% lignin, provides a suitable basis for the production of cellulose-rich materials with added value.

Previous studies have demonstrated the potential of agricultural residues as precursors for cellulose-based products. Materials such as microcrystalline cellulose, nanocellulose, cellulose nanofibers, and reinforced composites have been successfully produced from wheat straw, rice straw, sugarcane bagasse, and Miscanthus biomass. For example, Singh et al. showed that ultrasound-assisted alkaline treatment enhances cellulose recovery and improves the structural quality of the extracted fibers. Qu et al. reported the preparation of highly dispersed nanofibrillated cellulose from wheat straw using TEMPO-mediated oxidation, highlighting the benefits of surface functionalization. In addition, Hafid et al. found that chemical modification and controlled hydrolysis contribute to higher crystallinity and improved thermal resistance of cellulose obtained from biomass sources.

Cellulose remains one of the most widely investigated bio-based polymers because it combines mechanical strength with low density, biodegradability, biocompatibility, and thermal stability. These characteristics make cellulose an attractive reinforcing component for polymer composites and film-forming materials. However, direct incorporation of cellulose into polymer matrices does not always provide optimal performance because of its hydrophilic character and limited interfacial compatibility with certain polymers. To address this issue, a variety of modification strategies have been proposed.

Among the available approaches, TEMPO-mediated oxidation has received considerable attention due to its ability to selectively introduce carboxyl groups onto the cellulose surface under relatively mild reaction conditions. The resulting surface modification improves fiber dispersion and strengthens interactions between cellulose and polymer matrices. As a consequence, composites containing TEMPO-modified cellulose often exhibit enhanced mechanical behavior, greater thermal stability, and improved overall material performance.

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Polyvinyl alcohol (PVA) has been extensively used for the preparation of polymer films because of its transparency, flexibility, good chemical stability, and ease of processing. Owing to these characteristics, PVA is frequently selected as a matrix material for bio-based composites. Previous studies have demonstrated that the incorporation of cellulose fibers or nanocellulose into PVA can improve mechanical performance, thermal resistance, dimensional stability, and barrier behavior. Such improvements are generally attributed to the formation of stronger intermolecular interactions and a more compact composite structure.

Beyond their conventional applications, cellulose-reinforced polymer systems have recently attracted interest for radiation-protection purposes. A denser internal network and improved structural uniformity may contribute to greater attenuation of ionizing radiation, thereby enhancing the protective performance of the material. Despite the growing body of research on cellulose-based films and biodegradable composites, studies specifically addressing radiation-resistant materials derived from agricultural biomass remain relatively scarce. In particular, the potential of wheat-straw-derived cellulose as a reinforcement phase in sustainable radiation-shielding films has not yet been fully investigated.

Against this background, the present work explores the conversion of wheat straw into a value-added functional material with radiation-protective properties. Cellulose was isolated through alkaline delignification followed by bleaching and subsequently modified using TEMPO-mediated oxidation. The modified cellulose was incorporated into a PVA matrix to produce biodegradable composite films. Their structural, morphological, thermal, mechanical, optical, and radiation-shielding characteristics were examined using FTIR, XRD, SEM, TGA, UV–Vis spectroscopy, tensile testing, and gamma-ray attenuation analysis. By combining agricultural waste valorization with advanced composite design, this study contributes to ongoing efforts aimed at developing environmentally sustainable materials for radiation protection. The obtained results may support future applications in medical packaging, protective coatings, aerospace technologies, and other areas where lightweight and eco-friendly shielding materials are required.

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MATERIALS AND METHODS

Materials

Wheat straw obtained from agricultural fields in Uzbekistan served as the lignocellulosic precursor for cellulose production. Before use, the collected straw was washed thoroughly with distilled water to eliminate soil residues and other surface contaminants and then air-dried at room temperature. Polyvinyl alcohol (PVA, molecular weight 89,000–98,000 g mol^{-1}) was selected as the polymer matrix. Sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO), sodium bromide (NaBr), sodium hypochlorite (NaClO), and glycerol were obtained in analytical grade and employed as received. Distilled water was used in all experimental stages.

Extraction of Cellulose from Wheat Straw. The dried straw was milled and sieved to obtain particles below 500 μm. Cellulose isolation involved two consecutive treatments: alkaline delignification and peroxide bleaching. During the first step, the straw powder was treated with 10 wt.% NaOH solution at 90°C for 4 h under continuous stirring. This treatment facilitated the removal of lignin, hemicellulose, waxes, and other non-cellulosic constituents. The resulting solid fraction was washed repeatedly with distilled water until a neutral pH was reached.

The delignified material was subsequently bleached in a 5 wt.% H_2O_2 solution at 80°C for 2 h. The bleaching stage reduced the residual lignin content and increased cellulose purity. After completion of the treatment, the material was rinsed with distilled water until neutral conditions were obtained and then dried at 60°C in a convection oven to constant mass. The purified cellulose was preserved in sealed containers prior to further processing.

TEMPO-Mediated Oxidation. Surface functionalization of cellulose was achieved through TEMPO-mediated oxidation. A 1 wt.% cellulose suspension was prepared in distilled water and stirred until a uniform dispersion was obtained. TEMPO (0.1 mmol g^{-1} cellulose), NaBr (1 mmol g^{-1} cellulose), and NaClO (5 mmol g^{-1} cellulose) were introduced

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sequentially into the suspension. The oxidation reaction proceeded at 25°C for 4 h while maintaining the pH at 10.0 through controlled addition of NaOH solution.

Following oxidation, the suspension was repeatedly washed and centrifuged to remove residual reagents. The modified cellulose was then dried and stored for composite fabrication. The treatment generated carboxyl groups on the cellulose surface, which enhanced dispersibility and promoted stronger interactions with the polymer matrix.

Preparation of Composite Films. Composite films were fabricated using a conventional solution-casting approach. A 10 wt.% PVA solution was first prepared by dissolving the polymer in distilled water at 90°C under continuous stirring. TEMPO-modified cellulose was incorporated into the PVA solution at different loadings to produce four formulations: PVA-0C, PVA-10C, PVA-20C, and PVA-30C, corresponding to 0, 10, 20, and 30 wt.% cellulose, respectively.

Glycerol was added at 20 wt.% of the polymer content to improve flexibility and reduce brittleness. The mixtures were homogenized through mechanical stirring followed by ultrasonic treatment to ensure uniform cellulose distribution throughout the matrix. The resulting suspensions were cast onto glass substrates and dried at 40°C for 24 h. After drying, the films were removed from the glass plates and conditioned under ambient laboratory conditions before characterization.

Characterization. Chemical structure was investigated using Fourier Transform Infrared Spectroscopy (FTIR) within the spectral range of 4000–500 cm^{-1}. Crystallinity and phase characteristics were analyzed by X-ray Diffraction (XRD) over a diffraction range of 2θ = 5–60°. Surface morphology and cellulose dispersion were examined using Scanning Electron Microscopy (SEM) operated at an accelerating voltage of 5 kV.

Thermal behavior was evaluated by Thermogravimetric Analysis (TGA) from 30 to 700°C under a nitrogen atmosphere. Mechanical performance, including tensile strength, elongation at break, and Young's modulus, was measured using a universal testing machine in accordance with ASTM standards. Optical properties and ultraviolet light transmission were assessed by UV–Vis spectroscopy.

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Radiation-shielding capability was examined using a cobalt-60 (Co-60) gamma-ray source. The linear attenuation coefficient and transmission behavior were calculated from the measured gamma-ray intensities before and after penetration through the film samples. Each experiment was conducted three times, and the reported values correspond to the arithmetic mean of the measurements.

RESULTS AND DISCUSSION. The compositional changes observed after chemical treatment confirm the effectiveness of the cellulose isolation strategy. Comparison of the untreated biomass with the processed material (Table 1) reveals a marked reduction in hemicellulose and lignin content following alkaline delignification and peroxide bleaching. The progressive removal of these amorphous constituents increased the relative proportion of cellulose within the solid fraction, indicating successful purification of the lignocellulosic matrix. Such compositional modification is important because residual lignin and hemicellulose may negatively affect interfacial interactions, structural uniformity, and the overall performance of cellulose-based composites. The purified cellulose obtained in this study therefore provides a suitable reinforcement phase for the fabrication of biodegradable PVA composite films.

Chemical composition of untreated wheat straw and purified cellulose obtained after extraction. Table 1.

№	Component	Raw wheat straw (%)	Extracted cellulose (%)
1.	Cellulose	38.4 ± 0.8	91.3 ± 1.2
2.	Hemicellulose	28.7 ± 0.6	4.8 ± 0.3
3.	Lignin	19.5 ± 0.5	3.2 ± 0.2
4.	Extractives and ash	13.4 ± 0.4	0.7 ± 0.1

A pronounced shift in biomass composition occurred after alkaline delignification and peroxide bleaching. Cellulose content increased from 38.4 ± 0.8% to 91.3 ± 1.2%, whereas hemicellulose and lignin were reduced to 4.8 ± 0.3% and 3.2 ± 0.2%, respectively.

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In addition, extractives and ash were almost completely removed, with a residual content of only 0.7 ± 0.1%.

The final cellulose yield of 41.8% suggests that the purification procedure effectively removed undesirable constituents while preserving a considerable fraction of the structural polysaccharide. The decrease in lignin and hemicellulose is particularly important because these components are commonly associated with lower accessibility of cellulose surfaces and weaker interfacial interactions in composite materials. Consequently, the highly purified cellulose obtained in this study provides a favorable basis for subsequent surface modification and incorporation into PVA-based radiation-resistant films.

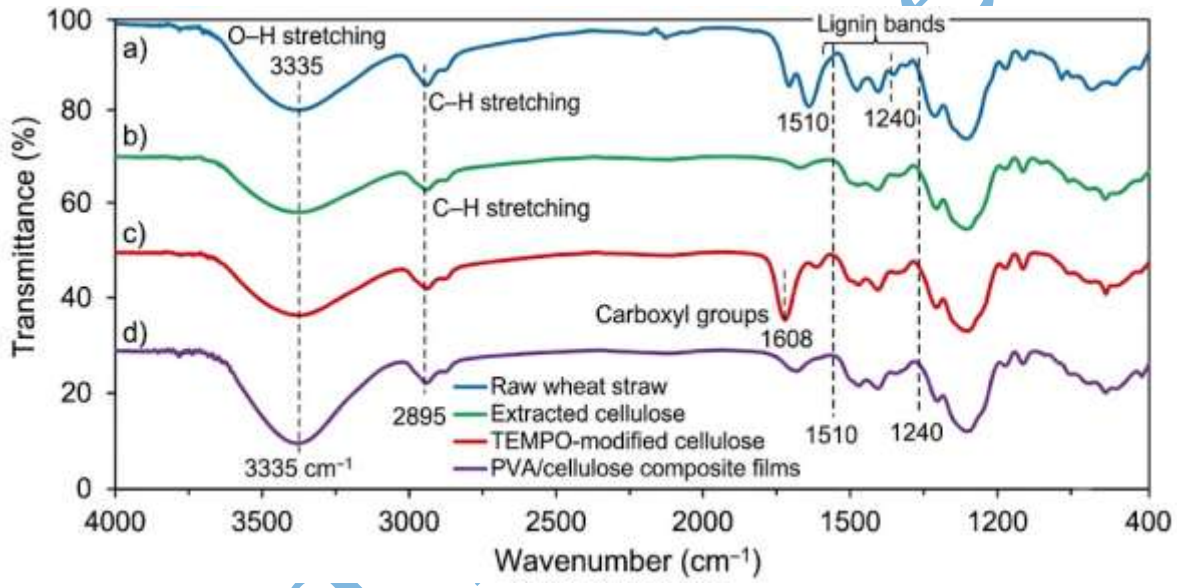

Figure 1. FTIR spectra illustrating the chemical changes occurring during cellulose extraction, TEMPO-mediated oxidation, and composite film fabrication.

The FTIR spectra provide evidence of the chemical transformations occurring during cellulose purification and subsequent TEMPO-mediated modification. In comparison with the untreated biomass, the absorption bands typically associated with lignin at approximately 1510 and 1240 cm^{-1} were no longer observed after the delignification and bleaching stages, indicating substantial removal of lignin from the lignocellulosic structure. A broad band centered near 3335 cm^{-1} was assigned to O–H stretching vibrations, whereas

the signal at 2895 cm^{-1} originated from C–H stretching vibrations commonly present in cellulose-based materials.

An additional absorption band appeared at 1608 cm^{-1} following TEMPO oxidation. This band is associated with carboxylate groups generated during the oxidation process and confirms the successful surface functionalization of cellulose. The stronger hydroxyl-related absorption observed in the composite samples suggests the development of intermolecular interactions between cellulose and the PVA matrix. Such interactions are likely to promote improved interfacial adhesion, which may explain the enhanced mechanical strength and thermal stability observed for the cellulose-reinforced films.

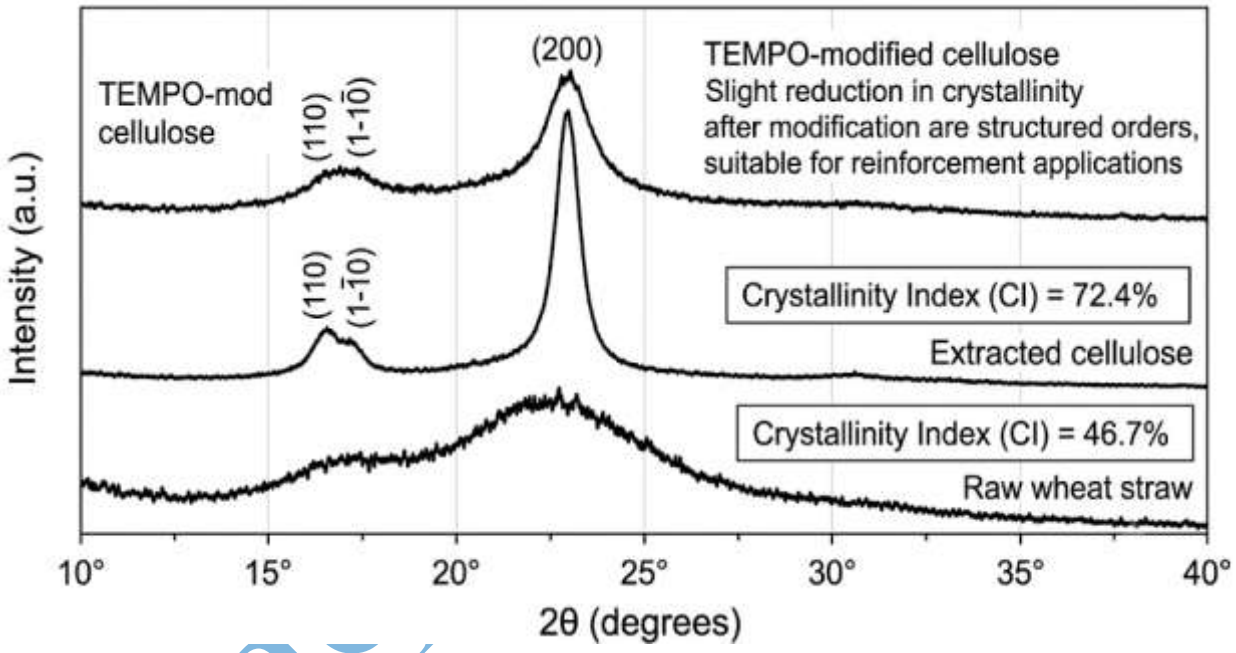

Figure 2. XRD profiles illustrating structural and crystalline changes in wheat straw during cellulose extraction and TEMPO-mediated oxidation.

The XRD analysis provides insight into the changes in cellulose structure occurring during purification and TEMPO-mediated oxidation. The diffraction pattern of raw wheat straw was characterized by relatively broad peaks, reflecting the presence of amorphous constituents such as lignin and hemicellulose within the lignocellulosic matrix. Following alkaline delignification and bleaching, distinct diffraction peaks appeared at approximately 2θ = 16° and 22.5°, which are characteristic of cellulose I. The emergence and sharpening

of these peaks indicate the removal of a substantial portion of the amorphous components and a corresponding increase in the relative crystalline fraction.

The calculated crystallinity index increased from 46.7% in untreated wheat straw to 72.4% in the purified cellulose sample. This increase is consistent with the elimination of non-cellulosic constituents during chemical treatment. After TEMPO oxidation, a small decrease in crystallinity was observed. Such behavior may be related to the introduction of surface carboxyl groups and limited disruption of crystalline regions during oxidation. Despite this reduction, the oxidized cellulose maintained a high degree of structural order, suggesting that the modification process preserved the crystalline framework required for effective reinforcement in composite materials.

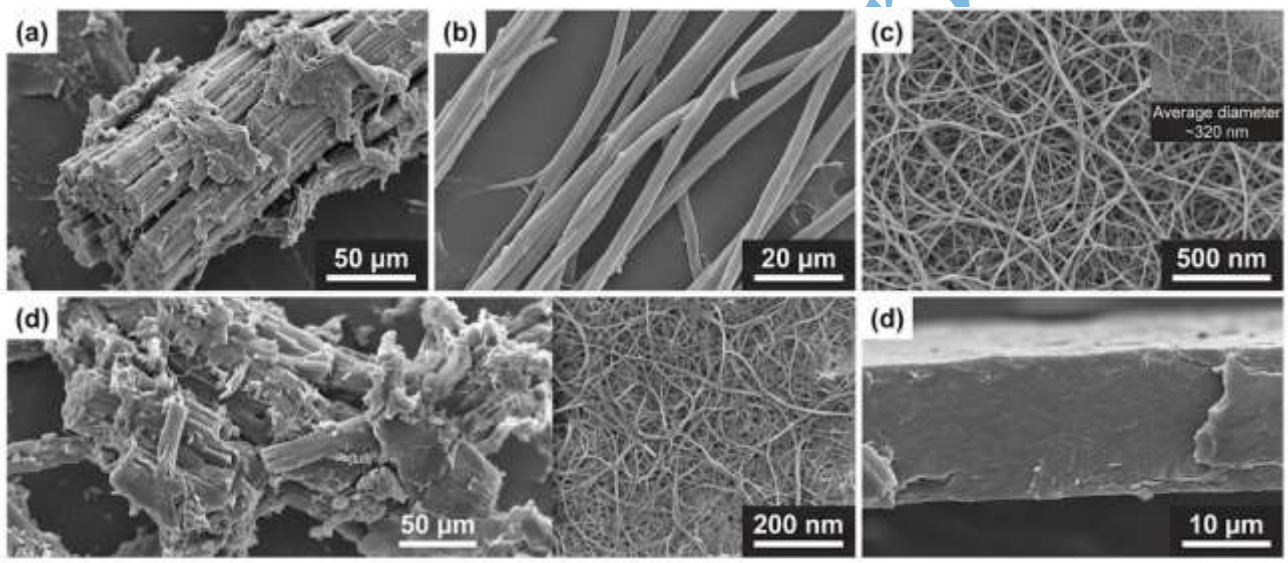

Figure 3. SEM micrographs of (a) raw wheat straw, (b) extracted cellulose, (c) TEMPO-modified cellulose, and (d) PVA-30C composite film.

SEM observations highlight the progressive morphological changes occurring during cellulose isolation and modification. The surface of the untreated wheat straw was characterized by densely packed fiber bundles together with surface deposits associated with lignin, hemicellulose, and other non-cellulosic constituents. After alkaline delignification and bleaching, the fiber structure became more distinct, reflecting the removal of the surrounding amorphous matrix and the exposure of cellulose microfibers.

Additional structural changes were observed after TEMPO-mediated oxidation. The treated cellulose exhibited a more individualized fibrillar morphology, forming an interconnected network with an average fiber diameter of approximately 320 nm. This behavior is consistent with the introduction of surface functional groups that facilitate fiber separation and improve dispersibility.

The microstructure of the PVA-30C composite film showed a dense and continuous morphology with no evident cellulose agglomerates. The absence of large aggregates suggests that the modified cellulose was distributed relatively uniformly throughout the polymer matrix. Such microstructural characteristics are generally associated with improved stress transfer between the reinforcement phase and the surrounding polymer, which may contribute to the enhanced mechanical performance of the composite films.

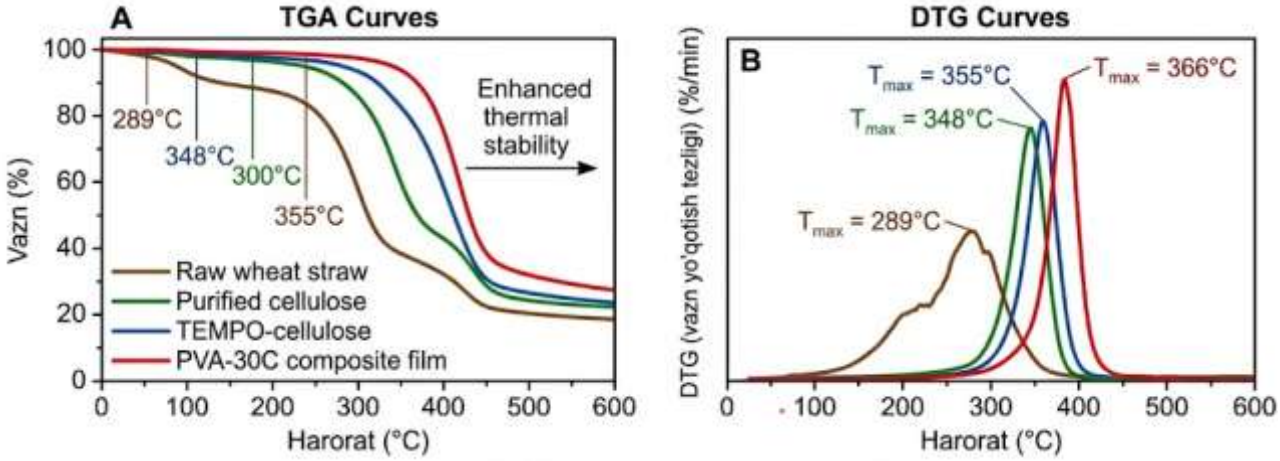

Figure 4. Thermogravimetric (TGA) and derivative thermogravimetric (DTG) curves of raw wheat straw, cellulose, TEMPO-modified cellulose, and PVA/cellulose composite films.

The thermogravimetric results indicate a progressive improvement in thermal stability following cellulose purification and composite film formation. Raw wheat straw exhibited a maximum degradation temperature (Tmax) of 289°C, which is consistent with the presence of hemicellulose and lignin components known to decompose at relatively lower temperatures. Removal of these constituents during chemical treatment resulted in a

noticeable shift of the degradation peak toward higher temperatures, with purified cellulose reaching a Tmax of 348°C.

A further increase in thermal stability was observed for the PVA-30C composite film, which exhibited a Tmax of 366°C. This behavior suggests that the incorporation of cellulose influences the thermal degradation pathway of the polymer matrix. The improved resistance to thermal decomposition may be associated with intermolecular interactions between cellulose and PVA, particularly hydrogen bonding, which can reduce chain mobility and increase the energy required for degradation. The observed increase in Tmax therefore reflects the beneficial role of cellulose reinforcement in enhancing the thermal performance of the composite films.

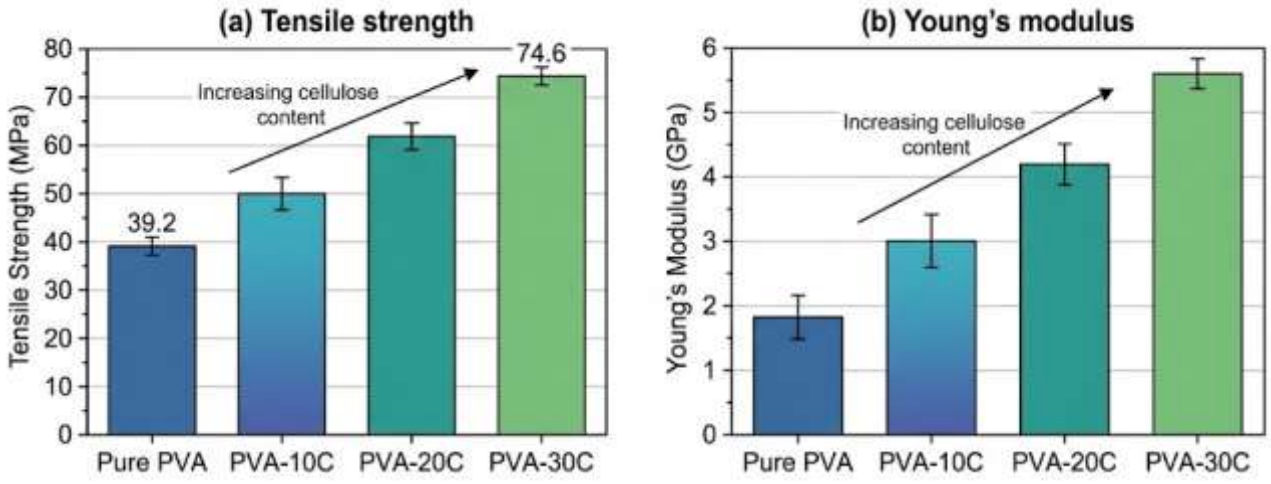

Figure 5. Mechanical properties of neat PVA and cellulose-reinforced composite films: (a) tensile strength and (b) Young's modulus.

The mechanical properties of the films were strongly influenced by the incorporation of TEMPO-modified cellulose. Tensile strength increased from 39.2 MPa for neat PVA to 74.6 MPa for the PVA-30C sample, indicating a substantial improvement in the load-bearing capacity of the composite system. A similar trend was observed for Young's modulus, which increased with cellulose loading, reflecting a progressive increase in film stiffness.

The improvement in mechanical behavior is likely related to the reinforcing effect of the cellulose network within the polymer matrix. The presence of well-dispersed cellulose

fibrils can facilitate stress distribution throughout the composite and reduce the likelihood of localized deformation under applied load. In addition, intermolecular interactions between hydroxyl-rich cellulose surfaces and PVA chains may contribute to stronger interfacial bonding. These structural features collectively support the enhanced strength and stiffness observed for the cellulose-reinforced films. The results demonstrate that wheat straw-derived cellulose can function as an effective bio-based reinforcement for biodegradable polymer composites.

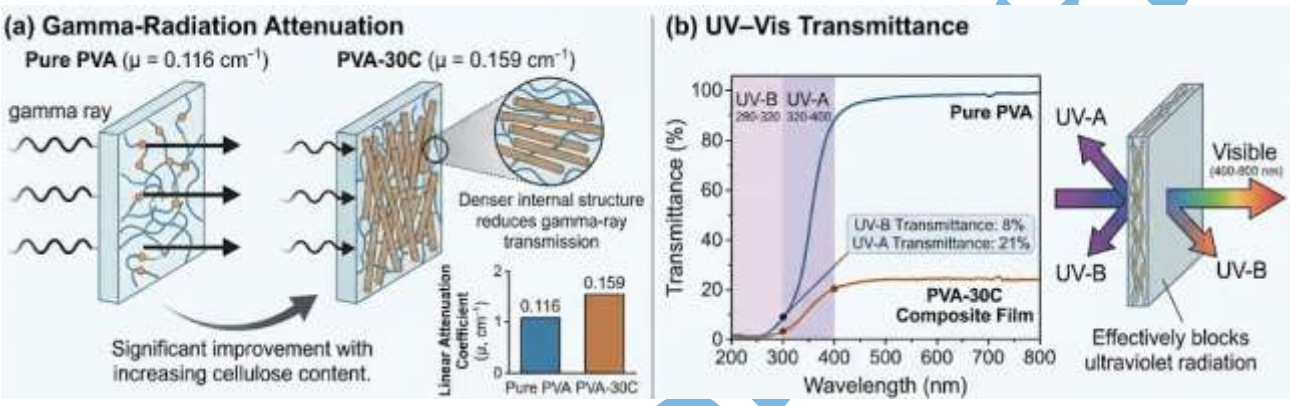

Figure 6. Radiation-shielding performance and optical transmittance of PVA/cellulose composite films.

The radiation-shielding characteristics of the films were influenced by cellulose incorporation. The linear attenuation coefficient increased from 0.116 cm^{-1} for neat PVA to 0.159 cm^{-1} for the PVA-30C formulation, indicating a reduction in gamma-ray transmission through the composite material. This behavior may be associated with the development of a more compact internal structure following the addition of cellulose. Such structural modification can increase the interaction probability between incident radiation and the material, thereby improving attenuation performance.

The UV–Vis spectra showed that the composite films also provided protection against ultraviolet radiation. The PVA-30C film exhibited UV-A and UV-B transmittance values of 21% and 8%, respectively, demonstrating a considerable reduction in UV transmission. The combination of radiation attenuation, UV-blocking capability, and

biodegradability suggests that these materials may be suitable for applications requiring lightweight and environmentally responsible protective films.

CONCLUSIONS. Cellulose with a purity above 90% was obtained from wheat straw through sequential alkaline delignification and bleaching. Subsequent TEMPO-mediated oxidation introduced surface functional groups that improved the compatibility of cellulose with the PVA matrix. Incorporation of the modified cellulose affected the structural, thermal, mechanical, and radiation-related properties of the resulting composite films.

Among the investigated formulations, the PVA-30C sample exhibited the highest thermal stability, with a maximum degradation temperature of 366°C, compared with 348°C for purified cellulose and 289°C for raw wheat straw. Mechanical testing revealed a substantial increase in tensile strength, from 39.2 MPa for neat PVA to 74.6 MPa for the cellulose-reinforced composite. Young's modulus also increased as the cellulose content increased, indicating greater rigidity of the composite structure.

Radiation-shielding performance improved with cellulose loading, as reflected by the increase in the linear attenuation coefficient from 0.116 cm^{-1} to 0.159 cm^{-1}. In addition, the composite films showed reduced transmission of ultraviolet radiation, with UV-A and UV-B transmittance values of 21% and 8%, respectively.

Overall, the results demonstrate that wheat-straw-derived cellulose can serve as an effective reinforcing component in biodegradable PVA composites. The combination of thermal stability, mechanical strength, radiation attenuation, and UV protection indicates potential applicability in areas such as protective coatings, medical packaging, and other material systems where environmentally sustainable shielding solutions are of interest.

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