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Mechanical and Morphological Enhancement of PVA Composites using *Crotalaria pallida* Fibers

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This study focuses on developing biodegradable films using Polyvinyl alcohol (PVA) as a matrix and *Crotalaria pallida* (CP) fibers, wood, and nanocellulose as reinforcing materials. Composite films with varying proportions of CP materials (0.5, 0.75, and 1.0g) were prepared using the solution casting method. The mechanical strength of the films improved markedly, with tensile strength increasing from 1.05 to 5.8 MPa in the PVAF3 composite, while elongation at break reduced from 95% to 57%, indicating increased stiffness. Fourier Transform Infrared Spectroscopy (FT-IR) revealed significant shifts in the –OH and –C=O regions, confirming hydrogen bonding and improved molecular interactions. Atomic Force Microscopy (AFM) indicated an increase in surface roughness (RMS) from 7.63 nm to 118.62 nm, suggesting enhanced fiber matrix interlocking. Thermogravimetric Analysis (TGA) demonstrated improved thermal stability, with decomposition temperatures rising from 200 °C to 300 °C. These biodegradable films have potential applications in sustainable packaging, biomedical devices, and agricultural films.

Keywords: Polymer composites; natural fibers; *Crotalaria pallida;* polyvinyl alcohol (PVA); mechanical properties

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The increasing use of non-degradable plastics has led to severe environmental concerns, with plastic production skyrocketing from 1.5 million metric tons in 1950 to 400 million metric tons currently [1]. Alarmingly, only 9% of plastic waste is recycled, while over 50% ends up in landfills and uncontrolled dumpsites. If current trends continue, plastic waste is expected to exceed 33 billion metric tons by 2050, with the World Economic Forum forecasting that plastic will outnumber aquatic life in freshwater bodies [2-3]. To mitigate this, developing sustainable, biodegradable materials is crucial.

Polyvinyl alcohol (PVA), a water soluble synthetic polymer, has garnered attention for its excellent film forming, emulsifying, and adhesive properties [4-5]. Its high tensile strength, flexibility, and biocompatibility make it suitable for various applications, including papermaking, textile sizing, and biomedical uses [6-8]. However, to further enhance its properties and sustainability, incorporating natural fibers can be beneficial [9-10].

Natural fibers, derived from renewable resources, offer benefits like biodegradability, low density, and improved mechanical properties [5, 8]. In this study, we utilized *Crotalaria pallida* fibers, a robust and woody fiber source from the Fabaceae family, as a reinforcing agent in PVA composite films. *Crotalaria pallida*, native to tropical and subtropical areas of India, has been traditionally used for various purposes, including medicinal applications [6, 11-12].

The incorporation of CP fibers into PVA can enhance the mechanical properties, thermal stability, and biodegradability of composite films [5]. The hydroxyl groups present in both PVA and CP fibers facilitate strong intermolecular hydrogen bonding, leading to improved compatibility and dispersion within the composite matrix [13]. This study focused on the development and characterization of PVA/CP composite films, exploring their molecular interactions, surface morphology, thermal, and mechanical properties [14].

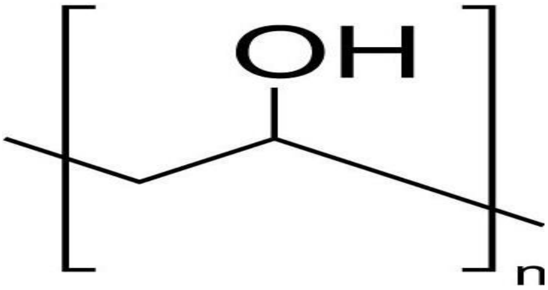


Figure 1. Structure of PVA.

For the first time, this research employs *Crotalaria pallida* fiber, along with wood and nanocellulose, as reinforcing materials to improve the properties of PVA composite films. By adopting a solution casting method and controlled drying process, we aim to create sustainable, biodegradable films with potential applications in packaging, biomedical devices, and agricultural films.

EXPERIMENTAL

Chemicals and Materials

Polyvinyl alcohol (molecular weight: 60000-125000 g/mol, degree of polymerization: 1700-1800, and viscosity: 9-21cps), NaoH, and methanol were purchased from HiMedia Laboratories Pvt. Ltd.; 8-Anilinonaphthalene-1-sulfonic acid ammonium salt hydrate (ANS) was supplied by Sigma-Aldrich; and glycerol (99.9% pure) was obtained from Changshu Hongsheng Fine Chemical Co. Ltd. *Crotalaria pallida* (CP) fibers, wood and nanocellulose (mature, >90 days) were sourced from Devagiri, Kerala, India, Milli Q water was used for all the preparations.

Methods

Extraction Process of CP Plant Materials

The extraction of *Crotalaria pallida* plant fibers involves a three stage process. In the first stage, alkaline hypochlorite cold retting, air dried bast fibers are treated with 8% NaOH solution for 48 hours, followed by sonication and drying to remove lignin. In the second stage, acid detergent treatment, the alkaline hypochlorite treated fibers are immersed in an acid detergent to remove hemicellulose. In the final stage, carboxymethylation, the acid detergent treated fibers are powdered, suspended in a 70% isopropanol/water solution, neutralized with glacial acetic acid, rinsed, and refiltered to solubilize and modify the fiber surface.

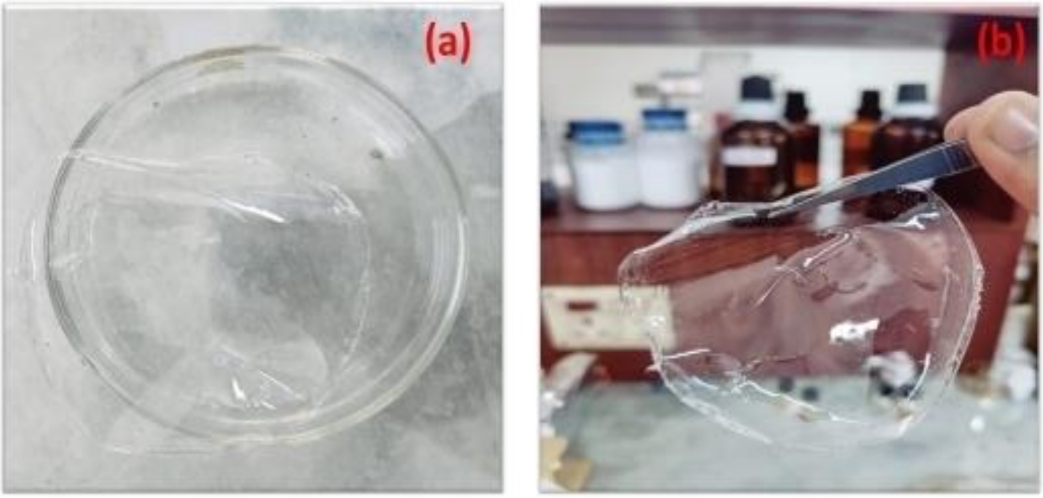


Figure 2. PVA film formation. (a) Wet film and (b) dry film.

Table 1. Compositions of PVA and PVA/CP films.

Sample no.	Sample name	PVA (4.0 w/v)	Glycerol (1.0 v/v)	*Crotalaria pallida* materials (g)	Final volume (ml)	Film formation (Yes/No)
1	PVA	4.0	1.0	0.00	30.0	Yes
2	PVAF1	4.0	1.0	0.50	30.0	Yes
3	PVAF2	4.0	1.0	0.75	30.0	Yes
4	PVAF3	4.0	1.0	1.00	30.0	Yes
5	PVAW1	4.0	1.0	0.50	30.0	Yes
6	PVAW2	4.0	1.0	0.75	30.0	Yes
7	PVAW3	4.0	1.0	1.00	30.0	Yes
8	PVAN1	4.0	1.0	0.50	30.0	Yes
9	PVAN2	4.0	1.0	0.75	30.0	Yes
10	PVAN3	4.0	1.0	1.00	30.0	Yes

PVA Composite Film Preparation

PVA and PVA/CP composite films were prepared using the solution casting method. A 4% PVA solution was made by dissolving 4 g of PVA in 100 ml of water and stirred continuously at 700-800 RPM and 70-80 °C for 4 hours. After forming a clear solution, 1% glycerol and 1 ml of 8-anilinonaphthalene-1-sulfonic acid ammonium salt hydrate (ANS) were added. The solution was cooled to room temperature, then cast into 15 × 10 cm silicon petri dishes. The dishes were placed in a drying chamber at 45°C for 24 hours. After drying, the films were removed from the petri dishes and stored in a dust free atmosphere until further studies.

Preparation of PVA Composites Containing CP Fiber, Wood, and Nanocellulose Materials

PVA solutions were added and stirred with different weight ratios of CP fibers, wood, and nanocellulose (0.5, 0.75, and 1.0 g). The solutions were stirred until the natural fibers were completely mixed with PVA. After mixing, the solutions were transferred into petri dishes (15 × 10 cm), allowed to dry at 40-45 ºC for 36 hours, and then maintained at RT for 24 hours. After being peeled off from the petri dishes, the films were used for characterization.

Characterization Methods

FT-IR Analysis

FT-IR analysis was used to determine molecular vibrations in the PVA and PVA/CP composite films. The FT-IR spectra of the films were obtained on a PerkinElmer 400 FT-IR spectrometer (IR version 10.6.0) equipped with an ATR attachment. A tiny film sample was placed on a "Golden Gate" diamond ATR and evaluated in the 4000-400 cm^{-1} range with a scanning resolution of 4 cm^{-1} and an interval of 1 cm^{-1} in an average of 16 scans.

Field Emission Scanning Electron Microscopy (FE-SEM)

Field emission scanning electron microscopy (FESEM) (TESCAN BRONO s.r.o., Czech Republic) was used to analyze the surface morphology. A tiny portion of the film was put on sticky carbon tape for SEM analysis. The carbon tape was then attached to an aluminum stud and vacuum-coated with gold. SEM images were captured at different magnification levels ranging from 15 to 20 kV.

Atomic Force Microscopy (AFM)

A confocal Raman microscope paired with an AFM (WITecALPHA 300RA) was used to evaluate the surface topography of the PVA and PVA/CP composite films. A silicon tip with a resonance frequency of 75 kHz and a force constant of 2.8 Nm^{-1} was used to obtain AFM photos during the tapping stage. The AFM pictures were captured with a scanning size of 10 × 10 micrometers and a radius of less than 8 nanometers. The WITec Control 4 application was used for processing and analysis.

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis of the PVA and PVA/CP composite films was performed using a PerkinElmer thermogravimetric analyzer (TGA 8000). According to the instrument's specifications, 4–5 mg of the sample was placed in a platinum crucible and heated at a flow rate of 50 ml/min in an inert nitrogen environment. DTG curves were used to find the maximal degradation temperature.

Water Solubility (WS)

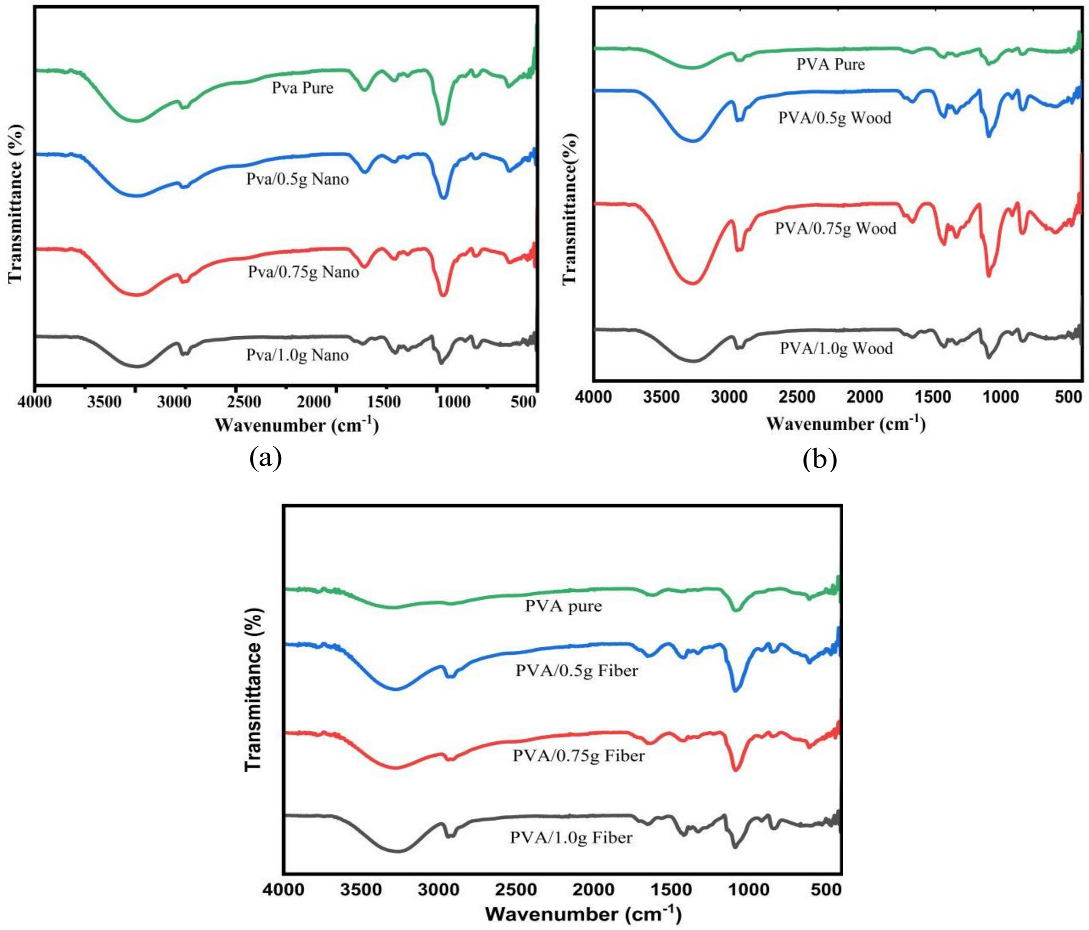
Water solubility (WS) is the proportion of a film's dissolved dry matter after immersion in water. The PVA and PVA/CP films were uniformly cut (10 mm × 10 mm), baked in an oven at 60°C for 5 hours, and then soaked in distilled water at room temperature for 24 hours. We removed the remaining samples and used tissue papers to remove any wetness. Next, we dried the materials at 100 °C for 5 hours before calculating the solubility. Finally, the WS was determined using the equation shown below.

$$\text{Water solubility (\%)} = (I_{dw}-F_{dw})/I_{dw} \times 100$$

I_{dw}= Initial dry weigh, F_{dw}= Final dry weight

Tensile Strength

A Universal Testing Machine (UTM) (AGS-X series 5 kN, Shimadzu) was used to determine the tensile strength. At room temperature, the tensile speed was set at 30 mm/min. Each sample was cut into 10 cm × 1.5 cm rectangles, and then tensile tests were conducted five times.



(a) (b)

(c)

Figure 3. FT-IR spectra of (a) PVA/CP nano, (b) PVA/CP wood, and (c) PVA/CP fiber.

RESULTS AND DISCUSSION

FT-IR Analysis

The FT-IR spectra of the PVA and PVA/CP composite films are shown in Figure 3. Pure PVA exhibits characteristic peaks at 3278 cm⁻¹ (O-H stretching), 2925 cm⁻¹ (asymmetric stretching vibration), 1636 cm⁻¹ (C=O), 1419 cm⁻¹ (CH bending), 1324 cm⁻¹ (CO stretching), 1081 cm⁻¹ (CO stretching), and 835 cm⁻¹ (O-H stretching vibration) [15]. The *Crotalaria pallida* spectrum shows similar O-H stretching (3200-3500 cm⁻¹), C-H stretching (2900 cm⁻¹), C=O stretching (1700-1730 cm⁻¹), C=C stretching (1500-1600 cm⁻¹), and C-O-C stretching (1000-1100 cm⁻¹) due to cellulose, hemicellulose, and lignin [15-16].

The FT-IR spectra of the PVA/CP nano and wood composites (Figures 3(a-b)) exhibit distinctive peaks in the range of 3550-674 cm⁻¹ [16]. The peaks at 3284 cm⁻¹, 2923 cm⁻¹, 1636 cm⁻¹, 1433 cm⁻¹, and 1071 cm⁻¹ for PVA/nano and 3270 cm⁻¹, 2931 cm⁻¹, 1650 cm⁻¹, 1417 cm⁻¹, and 1081 cm⁻¹ for PVA/wood indicate strong hydrogen bonding between PVA and CP materials [17]. The increased intensity of hydroxyl and C-O group peaks with rising CP content suggests good dispersion and compatibility between the matrix and CP materials [18].

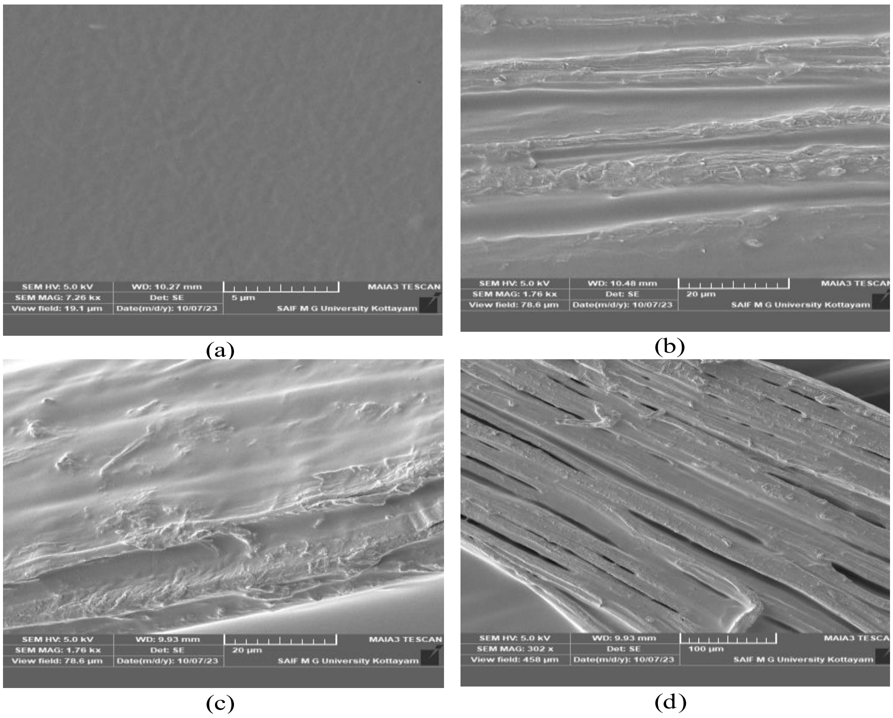
The FT-IR spectra of PVA with CP fibers are shown in Figure 3c. With increasing fiber content, the intensity of peaks slightly increases, indicating enhanced hydrogen bonding between PVA hydroxyl groups and cellulose in the fibers. The increased intensity at 3278 cm^{-1} confirms stronger hydrogen bonding, while changes in peak intensity at 1636 cm^{-1} and 1081 cm^{-1} suggest good interactions between fibers and matrix, improving overall cohesion [19-21].

FTIR analysis reveals that all three reinforcements, CP fibers, wood fibers, and CP based nanomaterials, enhance the molecular interaction with the PVA matrix, but to varying degrees. CP fibers improve hydrogen bonding and mechanical performance with moderate structural modification. Wood fibers exhibit stronger, bulkier interfacial interactions, contributing to enhanced matrix integration but with less uniform dispersion. In contrast, CP based nanomaterials provide the most defined and intense spectral shifts, indicating superior dispersion, surface reactivity, and molecular compatibility.

Scanning Electron Microscopy (SEM) Analysis

The surface and cross sectional morphology of the PVA and PVA/CP composite films were investigated using SEM. Pure PVA exhibits a flat surface, while the addition of CP fibers (0.5, 0.75, and 1.0 g) leads to a rougher texture with noticeable aggregates (Figure 4). The quantity and size of these aggregates increase with higher plant fiber contents. Despite this, the SEM images show no visible aggregation, cracks, or pores, indicating good component compatibility and a continuous matrix [22-23].

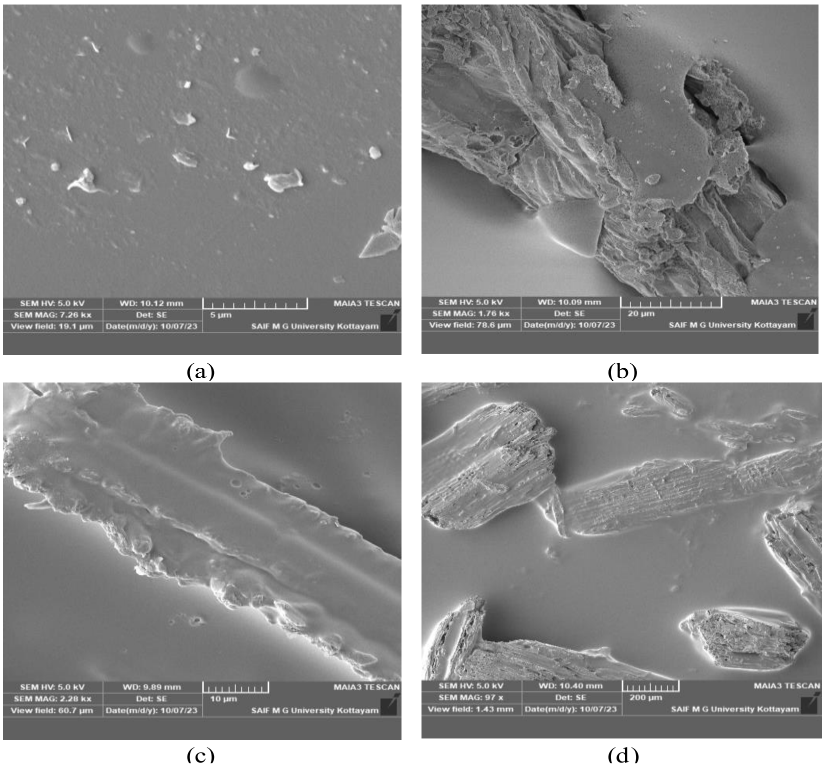
The morphology of CP nanocellulose particles is porous and irregular, with a rough surface (length: 170.7-409.9 µm; pore size: 5-45 µm) [24]. The addition of CP nanocellulose to PVA results in a rough surface with uniform distribution (Figures 5 (a-b)). Similarly, CP wood particles have an elongated plate-shaped morphology with rough edges (length: 182-646 µm) [24-26]. The PVA/wood fiber surface shows a homogeneous and rough appearance, with stronger intercalation between the PVA matrix and wood particles (Figures 5 (c-d)) [27-28].



(a) (b)

(c) (d)

Figure 4. SEM micrographs of (a) 5 µm pure PVA film, (b) 20 µm PVA /0.5g fiber, (c) 20 µm PVA/0.75 g fiber, and (d) 100 µm PVA/1.0 g fiber.



(a) (b)

(c) (d)

Figure 5. SEM micrographs of fracture surfaces of (a, b) 5, 20 µm PVA/CP nanocellulose films and (c, d) 10, 200 µm PVA/CP wood films.

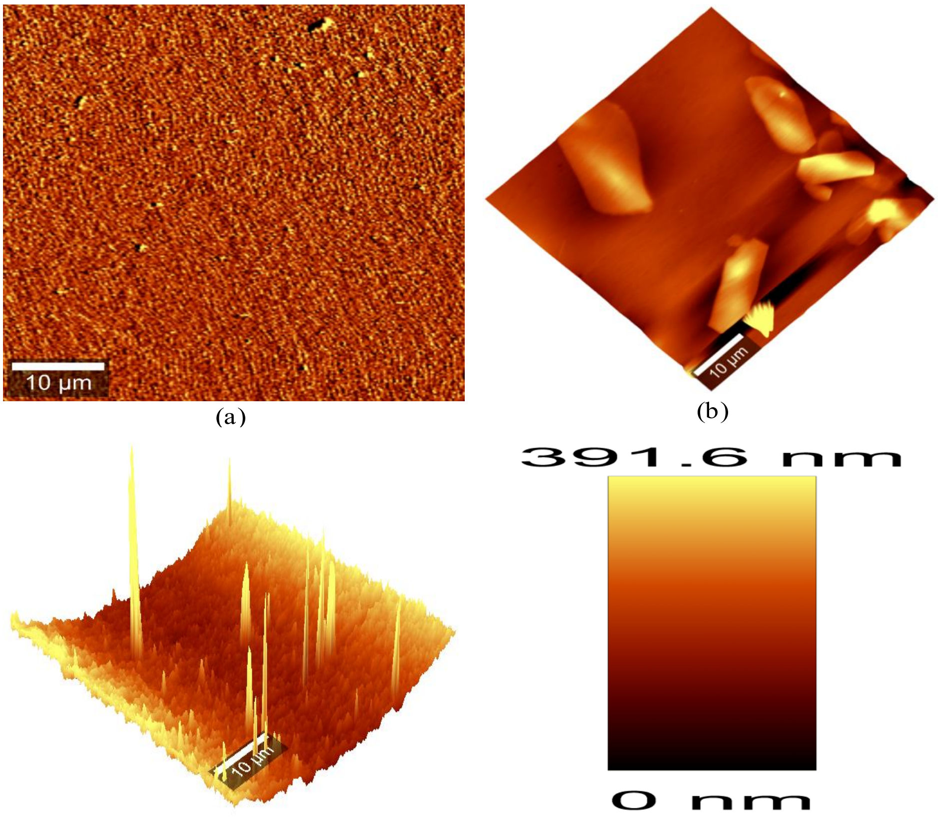
These findings suggest that plant fiber incorporation significantly affects the morphology of PVA membranes, influencing surface roughness and aggregate formation. The results indicate improved mechanical properties due to good dispersion and compatibility between the PVA matrix and plant fibers.

Atomic Force Microscopy (AFM) Analysis

AFM was used to investigate the surface morphology of the PVA and PVA/CP composite films. The 2D and 3D AFM images (Figure 6) show the surface morphology of films with varying CP fiber and nanocellulose contents. The root mean square (RMS) roughness values indicate that pure PVA has a smooth surface (RMS = 7.63 nm), while the addition of CP fibers and nanocellulose increases surface roughness. The RMS values follow the order: PVA < PVA/0.75 g CP nano < PVA/1.0 g CP nano < PVA/0.5 g CP fiber, with PVA/0.5 g CP fiber exhibiting the highest roughness (RMS = 118.62 nm) [27, 29].

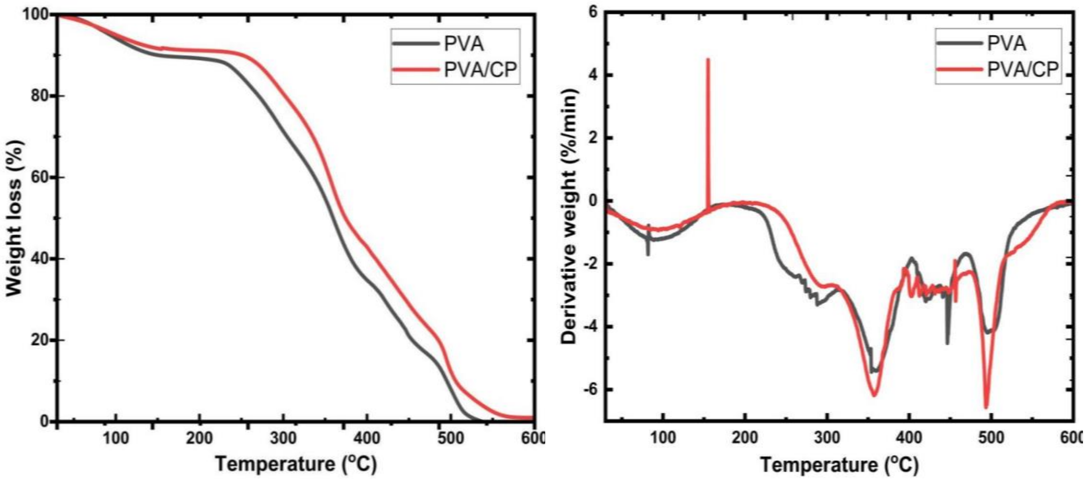
At higher concentrations, AFM images reveal dispersion and aggregation of PVA/fibers, leading to phase separation and a rougher surface. The increased surface roughness suggests polymer incompatibility and the formation of a heterogeneous phase, which is consistent with the SEM findings. Particle aggregation weakens the interaction between PVA and natural fibers, affecting the film's structural integrity.

The SEM and AFM analyses provide valuable insights into the surface morphology and topography of the PVA and PVA/CP composite films. The observed changes in surface roughness and morphology are crucial for determining the mechanical and thermal properties of the composites. The findings suggest that CP fibers/wood materials can be effectively used as reinforcement materials in PVA based composites.



(a) (b) (c) (d)

Figure 6. AFM images of (a) pure PVA Amplifier, (b) PVA/0.5 g fiber (2D), and (c, d) PVA/1.0 g nano (3D) and its peak height.



(a) (b)

Figure 7. TGA thermograms of (a) weight loss of PVA and PVA/CP fiber composite films and (b) derivative weight of PVA and PVA/CP composite films.

Thermogravimetric Analysis (TGA)

TGA and derivative thermogravimetric (DTG) analysis were conducted to investigate the thermal stability and degradation behavior of the pure PVA and PVA/CP fiber composites (Figure 7). The TGA curves reveal three primary stages of weight loss.

In the initial stage (100–200 °C), evaporation of residual moisture occurs, with a notable peak at approximately 150 °C for PVA/CP due to volatile compounds released from the CP fiber. The major degradation phase (250–450 °C) corresponds to the breakdown of polymer chains, where PVA/CP exhibits a broader degradation range, indicating a modified thermal degradation pathway. In the final decomposition stage (450–550 °C), the composite shows a higher char yield, demonstrating enhanced thermal stability as a result of CP fiber incorporation [28, 30].

DTG analysis confirms these observations, with distinct peak shifts and variations in decomposition rate. The composite materials exhibit lower peak intensity and a more gradual degradation pattern, suggesting improved thermal resistance [23, 31]. The presence of CP contributes to a more thermally stable structure, reducing polymer breakdown and increasing residual weight at higher temperatures. These findings suggest that CP reinforcement can improve the thermal properties of PVA based materials [19, 29].

Water Solubility (WS)

Water solubility is a pivotal property of polymeric materials, influencing their potential applications in biodegradable films, coatings, and water sensitive products. This study investigates the water solubility (WS) of polyvinyl alcohol (PVA) reinforced with wood and fiber, shedding light on the impact of these reinforcements on PVA's solubility characteristics.

Table 2. Water solubility of PVA based samples.

Sample no.	Sample name	WS (%)	Film thickness (mm)
1	PVA	16.69 ± 3.19	0.020
2	PVA/CP wood	14.00 ± 1.87	0.075 - 0.092
3	PVA/CP fiber	22.36 ± 3.127	0.090 - 0.105

WS of PVA and Its Composites

The WS values of pure PVA, PVA/wood, and PVA/fiber composites were determined to be 16.69, 14.00, and 22.36, respectively. The reduction in WS observed in PVA/wood composites can be attributed to the hydrophobic nature of wood, which restricts water absorption and dissolution. Conversely, the incorporation of fiber increased WS, suggesting that fiber's porous structure or hydrophilic nature enhances water interaction [31-32].

Implications for Biodegradable Applications

These findings have significant implications for the design of PVA based composites for biodegradable applications. By tailoring the type and content of reinforcement materials, it is possible to control the water solubility of PVA composites, thereby influencing their biodegradability and suitability for specific applications. For instance, PVA/fiber composites with enhanced water solubility may be suitable for applications requiring rapid biodegradation, such as agricultural films or disposable packaging materials.

Mechanical Properties: Tensile Strength Analysis

The tensile strength of PVA composite films reinforced with varying amounts (0.5, 0.75, and 1.0 g) of CP fibers and wood materials was investigated. The addition of fibers and wood increases tensile strength but decreases elongation. The results are presented in Table 3.

Tensile Strength Results

The tensile strength of pure PVA film was 1.05 MPa. The incorporation of CP fibers significantly improved tensile strength, with values of 2.4 MPa (0.5 g), 3.8 MPa (0.75 g), and 5.8 MPa (1.0 g). Similarly, PVA/CP wood composites exhibited tensile strengths of 2.3 MPa, 3.3 MPa, and 4.2 MPa for corresponding loadings [32]. In this study, the PVAF3 composite achieved a tensile strength of 5.8 MPa, which is significantly higher than neat PVA (1.05 MPa), indicating effective reinforcement through CP fiber. However, when benchmarked against published studies, the value remains moderate. For example, Asrofi et al. (2019) reported a tensile strength of 15.86 MPa for pure PVA and 12.94 MPa for PVA blended with 20% *bengkuang* (yam bean) starch. Similarly, Castro et al. (2023) observed tensile strengths up to 11 MPa for starch-based films reinforced with 10% sisal cellulose. These discrepancies may arise from differences in film thickness, PVA concentration, crystallinity, and interfacial compatibility [31, 33].

Table 3. Tensile strength of PVA composite films.

Sample no.	Sample name	Tensile strength (N/M^2)
1	PVA	1.05 ± 0.25
2	PVAF1	2.4 ± 0.19
3	PVAF2	3.8 ± 0.19
4	PVAF3	5.8 ± 0.16
5	PVAW1	2.3 ± 0.23
6	PVAW2	3.3 ± 0.20
7	PVAW3	4.2 ± 0.21

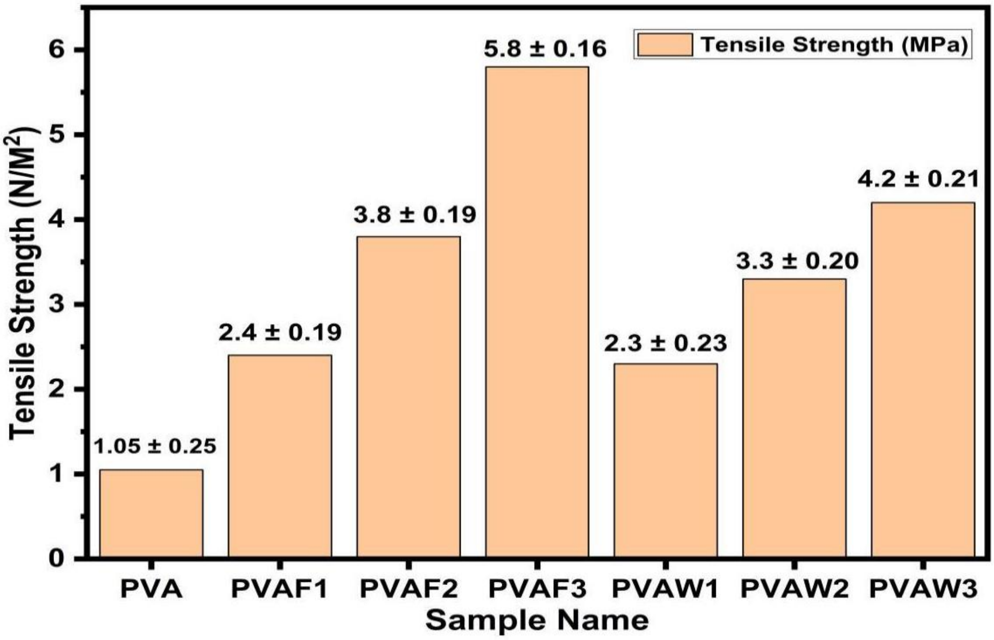


Figure 8. Tensile strength of PVA/CP fiber and PVA/CP wood.

Comparison of PVA/CP Fiber and Wood Composites

The tensile strength of the PVA/CP fiber composites was higher than that of the PVA/CP wood composites for all loadings. The maximum tensile strength was achieved in the PVA/CP fiber film due to excellent adhesion between the matrix and reinforcing material, ensuring effective stress transfer. The regular distribution of reinforcement materials inside the matrix ensures consistent mechanical properties throughout the film.

The study concludes that PVA/CP fibers possess higher tensile strength compared to wood, indicating favorable design, processing, and structural features. This suggests the suitability of PVA/CP fibers for applications requiring durability and reliability. The findings highlight the importance of reinforcement material selection and interfacial bonding in determining the mechanical properties of composite materials.

CONCLUSION

The study highlights the potential of CP fibers and wood materials as reinforcement materials in PVA composites. Incorporating *Crotalaria pallida* (CP) fibers into the PVA matrix significantly enhanced the biocomposite films' mechanical, structural, and functional properties. Tensile strength increased from 1.05 MPa for pure PVA to 5.8 MPa for PVAF3, while elongation at break decreased, indicating increased rigidity. FT-IR analysis showed intensified –OH and –C=O signals, confirming hydrogen bonding and improved molecular interactions. SEM revealed uniform fiber dispersion and strong interfacial adhesion with minimal fiber pull out in PVAF1. AFM indicated increased surface roughness (RMS: 7.63 nm to 118.62 nm), suggesting better fiber matrix integration. TGA showed enhanced thermal stability, with decomposition temperature rising from 200 °C (PVA) to 300 °C (composites). Water solubility decreased from 16.69% to 10.45% (PVAW3), reflecting improved moisture resistance. Overall, CP fiber reinforced PVA films demonstrate good mechanical strength, thermal durability, and moisture barrier properties, making them ideal for sustainable packaging and structural applications.

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