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POLYVINYL ALCOHOL/CHITOSAN BLENDING FILMS SUPPLEMENTED WITH *Crotalaria pallida* FIBERS: PREPARATION AND CHARACTERIZATION

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ABSTRACT

Eco-friendly composite films are used globally to minimize environmental effects and develop environmentally responsible products. In this study, we prepared and characterized composite films of polyvinyl alcohol (PVA) and chitosan (CS) reinforced with *Crotalaria pallida* (CP) plant materials. CP is a traditional medicinal plant from which we extracted different plant materials like fiber, wood, and nanocellulose and incorporated them with PVA/CS matrix. The incorporation of plant materials aimed to enhance the mechanical properties and biodegradability of the composite films. The solution casting process was used to prepare the PVA/CS films with a constant concentration of PVA and CS with different amounts of CP materials (0.5, 0.75, and 1.0 g). The films were characterized using Fourier transform-infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), atomic force microscopy (AFM), and tensile strength to evaluate their thermal, morphological, and mechanical properties. Among the different PVA/CS films loaded with CP materials fiber, wood, and nano, the films with CP fiber showed the best quality and strength. SEM and AFM images showed that the CP fibers were spread out evenly and stuck to the polymer matrix. The addition of CP fibers to the PVA/CS matrix resulted in the increase of tensile strength from 1.5 to 6.5 MPa. The prepared composite films have potential applications in biomedical, packaging, and agricultural fields due to their improved mechanical and biodegradable properties.

Keywords: Biodegradable Films, Polyvinyl Alcohol, Chitosan, *Crotalaria Pallida,* Mechanical Properties.
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INTRODUCTION

The increasing demand for sustainable and eco-friendly materials has led to the use of natural and biodegradable polymers. Biodegradable polymers offer a promising solution for environmental issues as they decompose naturally after their intended use.[1,2] They are widely used to make films, hydrogels, composites, and other specialized materials.[3] The polymer films are thin and flexible and are used for agricultural, packaging, and medical applications.[4] Polyvinyl alcohol (PVA) and chitosan (CS) are two biodegradable polymers that have gained attention due to their excellent biocompatibility, non-toxicity, and biodegradability.[5-8] PVA is a synthetic polymer known for its water solubility, flexibility, and good mechanical properties, making it suitable for a variety of applications, such as packaging, textiles, and biomedical.[9-12] PVA's properties, including chain length modulus, crystallinity range, and water solubility are influenced by its chemical structure, particularly hydrogen bonding among hydroxyl groups. PVA is widely used to make films owing to its remarkable properties like solubility, flexibility, and biodegradability. PVA films are a good oxygen barrier and resist grease, oils, and solvents.[13,14]

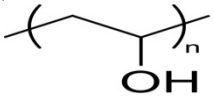

Fig.-1: Structure of PVA

CS is a naturally occurring polysaccharide formed from chitin, the second most common biopolymer after cellulose. Its notable features are biocompatibility, biodegradability, and antibacterial activity.[15,16] The outer skeleton of crabs contains chitin which deacetylates to form CS. The structure is made up of β-(1→4)-

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linked D-glucosamine and N-acetyl-D-glucosamine units that can be changed according to different medical and industrial needs. CS is also used for making films because of its bio-compatibility, biodegradability antibacterial nature, and oxygen barrier properties and found application in biomedical fields.[17-20]

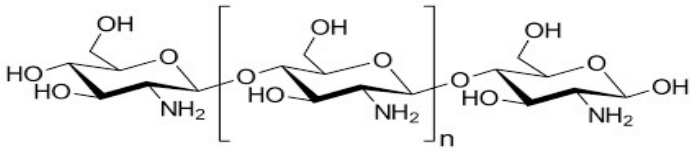

Fig.-2: Structure of CS

Even though PVA and CS form good films they have many limitations like water sensitivity and mechanical and thermal instability. However, their mechanical and thermal properties need improvement for various applications.[21-25] To address these problems researchers carried out many studies to prepare composite films by combining PVA/CS or incorporating other polymers, plant-based materials, nanoparticles, etc., into PVA/CS composite film.[26-29] Plant-based materials find more significance due to their lower environmental impact, biodegradability, and inherent antimicrobial properties.[30-31] *Crotalaria pallida* (CP) also known as smooth *crotalaria* is a nitrogen-fixing legume plant found in tropical Africa, Asia, and Australia. It is well known for its medicinal properties and role in improving soil fertility. The plant has bright yellow flowers and slender stems with blast phloem that provide strong woody fibers. The plant is widely used for medicinal and food purposes.[32-33] There are many works reported on the incorporation of plant materials into PVA/CS composite film, but to the best of our knowledge, there were no studies reported about CP plant material-reinforced PVA/CS films. In this work, we used CP-based materials like fiber, wood, and nano to enhance the properties of PVA/CS composite films. The solution casting method is used to prepare the PVA/CS/CP films in which the composition of PVA and CS is kept constant and different amounts of CP materials are added. The prepared films were examined using atomic force microscopy (AFM), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and Fourier transform infrared spectroscopy (FT-IR). The tensile strength studies were conducted to identify which CP plant material produces stronger films with PVA/CS.

EXPERIMENTAL

Materials

Polyvinyl alcohol (Molecular weight 60000-125000 g/mol, Degree of polymerization 1700-1800 and Viscosity 9-21cps), Chitosan (Degree of deacetylation is greater than 75%), NaoH, Methanol and Glacial acetic acid were purchased from HiMedia laboratories Pvt. Ltd. Glycerol purchased from Nice Chemicals Ltd., 8-Anilinonaphthalene-1-sulfonic acid ammonium salt hydrate (ANS) purchased from sigma-Aldrich and Ethanol (99.9% pure) purchased from Changshu Hongsheng fine chemicals Co. Ltd. *Crotalaria Pallida* (CP) plant stems were collected from Devagiri, Kerala, India. Milli Q water is used for all the preparations.

Extraction Process of CP Fibers

Cold retting of the CP stem with an alkaline hypochlorite solution is used to get the CP fibers. Air-dried bast fibers of CP are soaked in 8% NaOH solutions for 48 hours, then they are sonicated and washed, and methanol is added to get rid of the lignin. Secondly, the acid detergent retting process involves ground alkaline hypochlorite-treated plant material, filtered, washed, and dried for the removal of hemicellulose. The carboxymethylation process combines acidic detergent-treated ground plant fibers with isopropanol and water to modify their surface.

Preparation of Composite Films

The solution casting technique was used to produce the PVA/CS/CP composite films. 4% PVA solution is prepared by stirring for 4 hours at 70–80ºC. Simultaneously 2% chitosan solution is prepared in 1% (v/v) acetic acid solution by stirring for 2 hours at 60ºC. The PVA and CS solutions were mixed and added 4 ml of 1.0% (v/v) glycerol into it. The resulting solution was further stirred for 30 minutes at 60ºC to become homogeneous and sonicated for 15 minutes to remove air bubbles. The solution was divided into different portions with equal volumes and this added various amounts (0.5, 0.75, and 1.0 g) of CP plant materials (fiber, wood, and nano) and stirred for another 15 minutes to ensure uniform mixing. The resulting solutions

were then placed on silicon petri dishes and baked for twenty-four hours at 50°C. The composite films were peeled off from the Petri dishes and kept in a vacuum desiccator following the drying procedure. The samples were grouped into 4 distinct groups. PC represents PVA and CS, PCF1-PCF3 represents PVA and CS with CP fibers, PCW1-PCW3 represents PVA and CS with CP wood and PCN1-PCN3 represents PVA and CS with CP nano (Table-1).

Table-1: The Composition of PVA/CS with CP Materials

S. No.	Sample code	Nature of CP plant material	Amount of CP plant material
1	PC	-	-
2	PCF1	Fiber	0.50 g
3	PCF2	Fiber	0.75 g
4	PCF3	Fiber	1.00 g
5	PCW1	Wood	0.50 g
6	PCW2	Wood	0.75 g
7	PCW3	Wood	1.00 g
8	PCN1	Nano	0.50 g
9	PCN2	Nano	0.75 g
10	PCN3	Nano	1.00g

Fourier Transform-Infrared Spectroscopy (FT-IR)

FT-IR spectroscopy is used to identify molecular vibrations in PVA/CS and PVA/CS/CP composite films. A PerkinElmer 400 FT-IR spectrometer (IR version 10.6.0) equipped with an ATR attachment was utilized to acquire the FT-IR spectra of the samples. A tiny film sample was positioned on a "Golden Gate" diamond ATR and analyzed within the range of 4000-400 cm^{-1}, utilizing a scanning resolution of 4 cm^{-1} and an interval of 1 cm^{-1}, with an average of 16 scans conducted.

Thermogravimetric Analysis (TGA)

The thermogravimetric method analyzed the thermal deterioration of PVA/CS and PVA/CS/CP fiber films. The analysis was carried out by PerkinElmer (TGA 8000), which is from 30°C to 900°C temperature range and heating rate at 10°C/min. According to the instrument specifications, 4–5 mg of the sample was deposited in a platinum crucible and heated in an inert nitrogen atmosphere at a flow rate of 50 ml/min. We employed DTG curves to determine the maximum degradation temperature.

Field Emission Scanning Electron Microscopy (FE-SEM)

FE-SEM was used to study the morphology of the films prepared and the images were recorded using TESCAN BRONO s.r.o. Scanning electron microscope. The FE-SEM was recorded at 15-20 kV with a tiny sample deposited on the sticky carbon tape, which was then fixed on an aluminum stud and vacuum-coated with gold.

Atomic Force Microscopy (AFM)

The roughness and the RMS values of composite films were measured using a confocal Raman microscope coupled with an AFM (WITecALPHA 300RA). AFM images were taken in the tapping stage with a silicon tip at a resonance frequency of 75 kHz and a force constant of 2.8 Nm^{-1}. AFM images were captured with a scanning size of 30×30 micrometers and a radius of less than 8 nm. The AFM images obtained were processed and analyzed using the WITec Control 4 program.

Water Solubility (WS)

Water solubility (WS) refers to the percentage of the film's dissolved dry matter after water immersion. The PVA/CS and PVA/CS/CP films were cut to a uniform size (10 mm × 10 mm), dried in an oven at 60°C for 5 hours, then soaked in distilled water at room temperature for 24 hours. We collected the leftover sample and used tissue paper to remove the water. Next, we dried the sample at 100°C for 5 hours and calculated its solubility. We calculated the WS using the following equations:

Water solubility (%) = $(I_{dw}-F_{dw})/I_{dw}$ x 100

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

Tensile Strength (TS)

The Universal Testing Machine (UTM) (AGS-X series 5 kN, Shimadzu) was used to evaluate the tensile strength and elongation at break (EaB). The tensile speed was set at 30 mm/min at room temperature. Tensile tests were conducted five times for each sample after the films were cut into 10 cm × 1.5 cm rectangles.

RESULTS AND DISCUSSION

Preparation of Composite Films

PVA/CS/CP composite films were successfully prepared through the solution casting method. For the comparison of the effect of different CP plant materials (fiber, food, and nano), the composition and the amount of PVA and CS remained constant in all the films. The composite films were prepared by adding different amounts of CP fiber, wood, and nano (0.5, 0.75, and 1.0 g) and analyzed the properties through visual observations and the stretching process. The formation of composite films is shown in Fig.-3. The visual and stretching observations showed that PVA/CS films with CP fibers exhibited superior properties in terms of uniformity, strength, and quality. However, composite films with CP nano showed the weakest properties compared to fiber and wood with crack formation during peeling and stretching. It is also observed that the amount of plant material significantly influences the properties of the composite films with fiber and wood formed harder, rougher, and stronger films with the increase of plant content. In the case of CP nano, the reverse occurred, as the nano content increased, the formed films were weaker and cracked. From these observations, we can conclude that among the different CP plant materials, the use of fibers gave composite films with excellent properties.

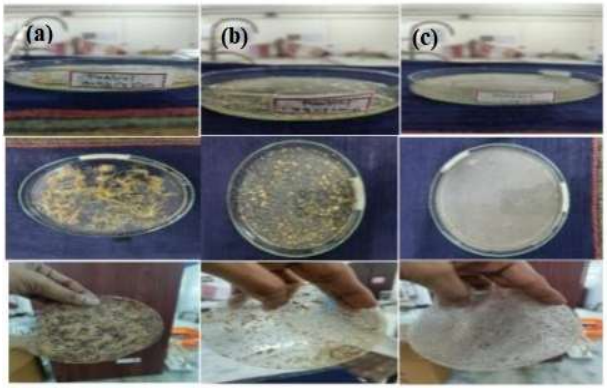

Fig.-3: Formation of PVA/CS/CP a) Fiber b) Wood c) Nanocomposite Films

FT-IR Results

FT-IR analysis has been used to investigate the structural changes of the different functional groups in the produced formulations. The FT-IR spectra of PVA/CS pure film and CP-incorporated films are shown in Fig.-4. The spectrum of pure PVA/CS film shows at 3283 cm^{-1} (OH group), 2925 cm^{-1} (C-H stretching), 1650 cm^{-1} (C=O stretching), 1459 cm^{-1} (CH-CH_2 stretching), 1053 cm^{-1} (C-O-C stretching), 853 cm^{-1} (C-C stretching) indicates the presence of PVA, CS.[5] Figure-4a demonstrates that the broad peak around 3200-3500 cm^{-1} attribution (O-H stretching vibrations) due to hydroxyl groups in PVA and chitosan indicates significant hydrogen bonding. These functional groups are represented by peaks in the spectra about 3272-3291 cm^{-1}.[6] The peak at ~2925 cm^{-1} is attributed to C-H stretching from aliphatic methylene groups in PVA or other components. The peak at 1650-1634 cm^{-1} is attributed to the amide band caused by chitosan's C=O stretching or water absorption. The addition of CP fiber indicated stronger peaks in PCF1, PCF2, and PCF3 than in PC. Peaks at 1431-1459 cm^{-1} (C-H bending vibrations) and 1067-1016 cm^{-1} (C-O stretching) were observed in PVA or chitosan saccharide structures. The peak at around 835-848 cm^{-1} indicates the occurrence of PVA-chitosan interactions. [7] The broad peak at 3200-3300 cm^{-1} (3270-3283 cm^{-1}) represents O-H stretching vibrations from hydroxyl groups in PVA, CS, and CP wood composites (fig.4b). The peak is broad and slightly shifted, implying hydrogen bonding. The shift and intensity shifts in PCW samples indicate interactions between wood components and the polymer matrix. Aliphatic methylene groups in PVA and wood have a peak of about 2920-2930 cm^{-1}, indicating C-H stretching vibrations The polymer backbone and wood fibers contribute, as evidenced by their consistent presence in all samples. The peak at

1650-1644 cm^{-1} indicates an amide band (C=O stretching) in chitosan or absorbed water in the matrix.[8] The peak around 1415-1416 cm^{-1} is due to C-H bending vibrations of the cellulose structure. PVA, glycosidic linkages in chitosan, or cellulose fibers exhibit a peak of about 1050-1047 cm^{-1}, indicating C-O stretching. The increased intensity in PCW samples represents the contribution of wood-based cellulose. The peak near 843-840 cm^{-1} is attributed to out-of-plane bending, most likely caused by the polymer network or cellulose additives. [9]

From Fig.-4c the broadband at 3200-3300 cm^{-1} indicates hydrogen bonding between PVA, CS, and CP nano, and the peaks at 2920 cm^{-1}, 2921 cm^{-1}, 2930 cm^{-1}, and 1434 cm^{-1}, 1427 cm^{-1}, 1420 cm^{-1} (PCN1, PCN2 and PCN3), suggest the presence of alkyl groups in PVA and CS. The peaks at 1635 cm^{-1}, 1635 cm^{-1}, 1640 cm^{-1},1150 cm^{-1}, and 1080 cm^{-1} confirm the presence of C-O and C-O-C groups in PVA and CS. The specific peaks at 1740 cm^{-1} and 1250 cm^{-1} indicate the presence of cellulose and hemicellulose in the CP nano. [10]

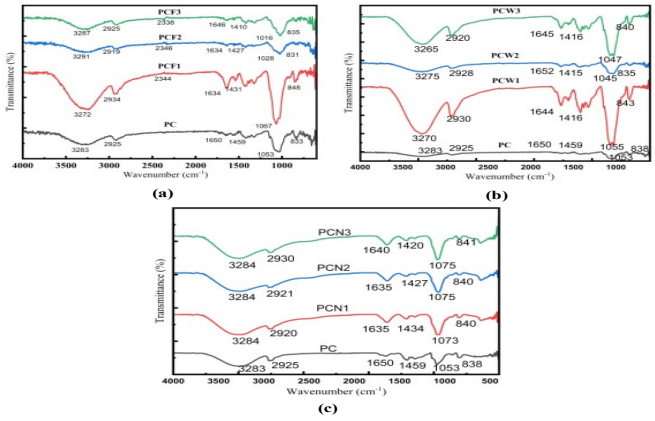

Fig.-4: FTIR Spectra of a) PCF1-PCF3 b) PCW1-PCW3 c) PCN1-PCN3

The hydroxyl peak's intensity increased with CP materials content, indicating improved hydrogen bonding between the fiber, wood, and nanocellulose with the polymer matrix. With CP addition, the C=O stretching peak shifted to lower wavenumbers, indicating improved fiber-matrix interaction and enhanced mechanical and thermal properties.

SEM Analysis

The morphology of PVA/CS pure film and CP incorporated films were analyzed through SEM and the SEM images of different films are shown in Fig.-5. For the comparison, the SEM images of different plant materials with the same quantity of plant material (0.5 g) are taken. The SEM images of PVA/CS film showed a homogeneous and smooth surface indicating the enhanced interaction between PVA and CS and forming a uniform polymer matrix. In the case of PVA/CS/CP fiber films showed a well-dispersed and uniform surface without any gaps or cracks which attributed to the good compatibility of CP fiber within the polymer matrix. [12,14]

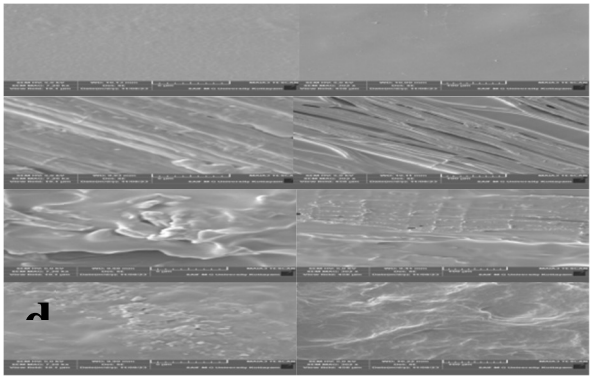

Fig.-5: SEM Images of PVA/CS a) Pure with b) CP Fiber c) CP Wood and d) CP Nano at different Magnifications (5 µm and 100 µm)

In the case of CP wood and CP nano, the surface showed an irregularity with visible gaps within the system. The SEM images support the visual observations, concluding fibers incorporated uniformly through the PVA/CS matrix.[16, 17] The morphology of PVA/CS pure film and CP incorporated film analyses through SEM and the SEM images of different films are shown in Fig.-6. The SEM images of PVA/CS films showed a homogeneous and smooth surface indicating the enhancing interaction between PVA and CS and forming a uniform polymer matrix. [18,20,21] In the case of PVA/CS/CP fiber films showed well-dispersed and uniform surfaces without any gaps or cracks which attributed to the good compatibility of CP fiber within the polymer matrix.

Water Solubility (WS)

In PVA/CS composite films, the water solubility test is usually done to see how much the films dissolve in water or how resistant they are to water. The test results can show how stable the film is, how well it absorbs water, and whether it is suitable for certain uses, like biodegradable packaging, medical uses, or drug delivery systems. From Table-3, the test indicates that pure PVA/CS film solubility is only 13.5%, which suggests that the equilibrium between hydrophilic and cross-linked regions is stable. In PVA/CS/CP fiber films, as CP content increases from PCF1 to PCF3, the water solubility rises due to the higher proportion of hydrophilic groups and reduced structural cohesion. [22] In PVA/CS/CP wood fibers, a slight increase in water solubility from PCW1 to PCW3 indicates the cross-linking or structural reinforcement of wood with a matrix. According to the results, PVA/CS/CP fiber films are suitable for applications requiring fast degradation or solubility, while PVA/CS/CP wood films are used for applications requiring water resistance and slower degradation.

AFM Results

The surface roughness of the film was measured using AFM. Surface roughness is an important parameter in many applications as it influences the properties and the performance of the material. AFM is commonly used to measure surface roughness, and the root mean square (RMS) value is one of the important parameters obtained from AFM measurements. [26] The low RMS value indicates the smoothness of the surface, and the high RMS value corresponds to a rougher surface. Even though the optimum roughness of the films is considered as an indication of good films, greater RMS values measure the greater roughness of the film which can lead to the quality loss of the film.

Table-2: Comparison of RMS Values of different PVA/CS Composite Films

Sample code	RMS value (nm)
PC	6.4
PCF1	118.0
PCF2	130.0
PCF3	212.0
PCW1	95.0
PCW2	101.0
PCW3	198.0
PCN1	79.0
PCN2	92.0
PCN3	103.0

The RMS value of PVA/CS film is very low compared to the other composite films indicating the formation of a smooth surface and it is by the SEM analysis. A comparison of RMS values of different composite films is shown in Table-2. From the table, it is clear that, compared to nano and wood, fiber showed more roughness and an increased amount of CP loading increased the roughness to a higher extent.

Tensile Strength Results

To use biodegradable films for applications like packaging materials, they should possess comparable mechanical properties. We measured the mechanical properties of the composite films, including tensile strength (TS) and elongation at break (EaB). TS outlines the highest level of stress a material can endure

when subjected to tension or stretching before failure. It measures the amount of force that a material can hold without failure.

Table-3: Physical and Mechanical Properties of PVA/CS Composite Films

Sample code	Water solubility (%)	Tensile strength (MPa)	Elongation at Break (%)
PC	13.5	1.5	75
PCF1	40.54	3.6	77
PCF2	47.15	4.9	60
PCF3	55.58	6.5	45
PCW1	13.67	2.9	50
PCW2	15.01	3.9	41
PCW3	16.77	5.1	30

EaB indicates the ability of a material to get stretched before breaking. This shows how long the material can withstand or undergo deformation before failure. The TS and EaB of the composite films with PVA and CS and PVA/CS with CP are shown in Fig.-6 and Fig.-7. It is noted that PVA/CS film has a TS of 1.5 MPa (Nm^{-2}) with increasing its TS on CP loading. In comparison to CP wood, fiber loading significantly increased the TS with the increase of its value on the increment of fiber loading. A similar trend is also observed for CP wood, concluding the fact that the amount of CP content increases the tensile strength despite its nature. [27] In the case of EaB, it is decreased as the CP content increases. However, CP fiber showed higher EaB in comparison to CP wood. Interestingly, pure PVA/CS film and PCF1 have almost similar EaB values, which suggests that fiber loading does not significantly affect the ability of films to deform before failure. This is attributed to the fact that the incorporation of plant fibers does not make the film brittle and has good compatibility between the polymer matrix and CP fiber.[28] The relationship between TS and EAB is inversely proportional in the case of prepared films. This means films with the highest TS will have the lowest value of EAB for both wood and fiber, showing the fact that they are hard to break and have lower flexibility. It is also important that since these parameters are changed with the amount of content inside the films they can adjust according to our application demands. But obviously, it is observed that the incorporation of CP fibers improves the mechanical properties of the films and it is a sustainable replacement of toxic and nondegradable materials.

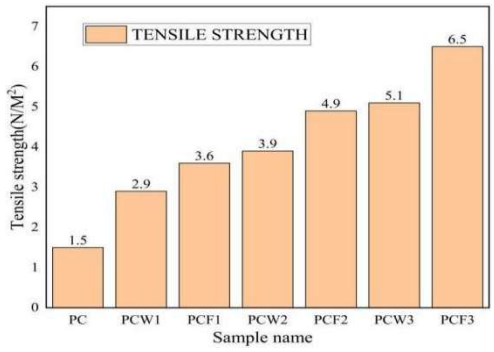

Fig.-6: Tensile Strength of Composite Films (PC, PCW1-PCW3, PCF1-PCF3)

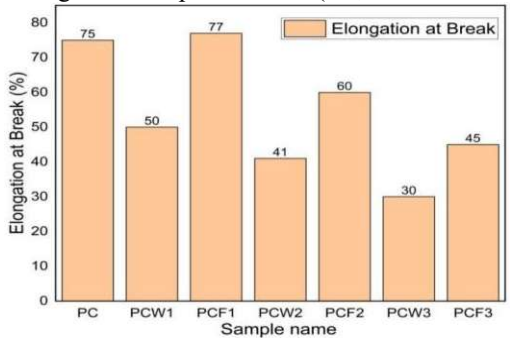

Fig.-7: Elongation at Break of Composite Films (PC, PCW1-PCW3, PCF1-PCF3)

Thermogravimetric Analysis (TGA)

We use TGA to determine the thermal degradation of the composite films at various temperature conditions. In this study, we analyze the PVA/CS and PVA/CS/CP fiber film degradation rates. Figure-5a illustrates the categorization of PVA/CS and PVA/CS/CP fiber film degradation rates into three distinct temperature intervals. The initial degradation of PVA/CS film was observed at 30–160 ºC due to water evaporation, and the second stage of weight loss is up to 390 ºC (46%) due to PVA breakdown and chitosan's saccharide structure. [29] The third degradation stage was seen up to 600 °C (34%), indicating the complete breakdown of PVA and chitosan. At temperatures below 160 °C, PVA/CS/CP fiber film has a slight weight loss, which could be attributed to moisture loss from the film. In comparison with the PVA/CS film, the weight loss during the second stage of degradation is smaller. This is because it involves the destruction of three parts: PVA, CS, and CP fibers, and the final degradation can be seen up to 600 °C (Fig.-8a). [30]

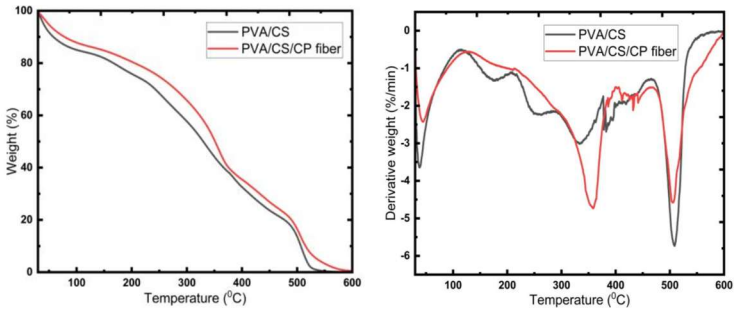

Fig.-8: TGA Thermograms of a) Weight Loss of PVA/CS and PVA/CS/CP Fiber Film and b) Derivative Weight of PVA/CS and PVA/CS/CP Fiber Films

It is evident that (Fig.-8b) the weight loss of PVA/CS film in all temperature conditions gradually increases and reaches above 500 °C; the maximum loss was observed. The PVA/CS/CP fiber film is more thermally stable than the PVA/CS film. This is because the CP fibers are spread out evenly, and there are strong intermolecular interactions that make the film more crystalline, which makes it more thermally stable.

CONCLUSION

The urge for sustainable alternatives to conventional plastic materials owing to their environmental challenges is a hot topic of research nowadays. Biodegradable films offer a promising replacement for plastic waste thereby reducing environmental problems. Polyvinyl alcohol (PVA) and chitosan (CS) are well known for their film-forming ability and are used for many applications including packaging, agriculture, the biomedical field, etc. The growing demand for biodegradable films also challenges the properties required to fit in a desirable application. This shows the significance of the incorporation of other materials into the existing films in which plant-based materials are highly focused on their economical and ecofriendly values. *Crotalaria pallida* (CP) is a well-known source of traditional medicine and woody fibers are exclusively used to modify PVA/CS composite films. In this work, we used CP-based different materials like fiber, wood, and nano out of which fiber-based PVA/CS films showed the best results in terms of quality and strength.

The work involves simple preparation and utilization of plant material, further enhancing the significance and scope of the study. As we look forward to the future there remains further scope for innovating and optimizing the material according to the application required and presents an opportunity to enhance the antimicrobial properties and bio-compatibility of the films. The shift towards biodegradable films with plant materials from traditional ones is not only a solution to environmental impacts but also makes sustainable materials with better performance eco-friendliness..

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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