

Characterization and Synthesis of Biocomposite Film with Coir and Polyvinyl Alcohol/Polyethylene Glycol

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Received 31 July 2023; revised 26 December 2023; accepted 15 January 2024

This study explores the synthesis of biodegradable composite films by incorporation of different amounts of polyethylene glycol with polyvinyl alcohol, untreated coir and treated coir using the solution casting method. The effect of polyethylene glycol on the structure and properties of the synthesized biocomposite films was investigated. The increased ratio of polyvinyl alcohol/polyethylene glycol resulted in decreased tensile strength. This investigation revealed that the incorporation of 10 wt% polyethylene glycol with polyvinyl alcohol was sufficient to synthesize the biocomposite film and resulted in tensile strength of 28.14 MPa. It was observed that the incorporation of 30 wt% polyethylene glycol led to phase separation with the tensile strength of 18.33 MPa and hence, 10 wt% polyethylene glycol incorporation is best among the tested treatments for synthesizing biodegradable composite film.

Keywords: Biodegradable composite film, Environmentally-friendly polymers, Natural fibers, Solution casting method, Tensile strength

Introduction

It has become more important to employ ecologically friendly materials due to increased environmental consciousness, community concern, updated environmental policies and unsustainable petroleum usage. Natural fiber stands out as a notably eco-friendly material with superior qualities compared to synthetic fiber.¹⁻³ Hence, natural fibers have become more prevalent in the world of composite materials due to their availability, recyclability and biodegradability.⁴⁻⁷ The presence of noncellulosic components (hemicellulose, lignin, pectin etc.) in the natural fiber hinders the bonding characteristic between the natural fiber and the matrix hence, it is desirable to remove these components to enhance the properties of the composite.^{8,9} Therefore, the fiber surface needs to be treated with various chemicals in order to improve compatibility between the fiber and the matrix material.¹⁰ Some of the treatment methods can be named as silane treatment (ST)¹¹, sodium chlorite treatment (SCT), acetylation treatment (ACT), isocyanate treatment (ICT), benzoylation treatment (BT)¹², alkaline treatment (AT)¹³⁻¹⁵, peroxide treatment¹⁶, stearic acid treatment (SAT) and permanganate treatment (PT).^{17,18}

Coir fibers are derived from the outer husk of the coconut. *Cocos nucifera* L. is the scientific name for the coconut tree, a tropical plant species belonging to the Areaceae family.¹⁹ Like other woody materials, coir fibers comprise cellulose, hemicellulose and lignin.²⁰

Polyvinyl alcohol (PVOH) is a synthetic thermoplastic polymer obtained through the hydrolysis of polyvinyl acetate.²¹ Strong intramolecular and intermolecular hydrogen bonds can form with multi-lateral hydroxyl groups, giving PVOH its high tensile strength, outstanding adhesive capabilities, non-toxic, excellent film formation, better biocompatibility²², abrasion resistance and anti-alkaline resistance.²³ Polyethylene glycol (PEG) is a commonly used polymer that exhibits extraordinary qualities such as solubility in many organic solvents, good thermal resistance, high chain flexibility, non-toxicity and an excellent capacity for absorbing water.²² To create films with superior mechanical and thermal properties, PVOH has also been blended with PEG, chitosan, starch, alginate and tara gum.^{21,24} However, not much work has focused on the effect of PEG with PVOH in synthesizing biodegradable composite film. Miscible blends between PVOH and PEG polymers are formed by the hydrogen bonding (–H) interaction of the hydroxyl groups (–OH) of PVOH with the ether linkage present in PEG chains.²⁵

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In this study, polymer films are synthesized by varying the amount of PEG in PVOH/PEG films and the natural fiber-based polymer films are fabricated by integrating untreated and alkali-treated coir fiber with PVOH-PEG blend. Therefore, in the present study, we have observed the effect of PEG in the fabrication of biodegradable composite films.

Materials and Preparation Methods

Materials

Coir was collected from the coconut's outer husk and was procured from the local vendors near the Durga Kund temple in Varanasi, Uttar Pradesh, India. Polyvinyl alcohol (PVOH) from HiMedia, 86%–89% hydrolyzed and polyethylene glycol (PEG) 6000 were purchased from Merck.

Methods

Coir was cut into pieces 2.5 cm long and cleaned adequately with distilled water. Sun-exposed drying was done for 5 to 7 days to reduce the involvement of electricity. It was then dried in an oven at a temperature of 70°C for one day to remove any remaining moisture. Coir was ground to get the powdered form of coir fiber and sieved with the sieves of mesh no. 120.

Alkaline Treatment

In the 400 mL of 5% sodium hydroxide (NaOH) solution, 35 g of powdered coir fibers were soaked. The solution was then gradually heated with a magnetic stirrer for 55 minutes at 75°C at 450 rpm. The coir fibers were then washed with distilled water using a vacuum filtration process until a neutral pH was attained. The washed coir fibers were thoroughly dried completely in an oven at a temperature of 70°C. Further, dried coir fibers were ground into a powder using a grinder and sieved through sieves of mesh no. 120.

Fabrication Technique

The Solution casting technique was adopted to fabricate the composite films, as previously reported by Srivastava *et al.*²⁶, with minor modifications. The untreated coir fiber (UCo), treated coir fiber (TCo), PVOH and PEG were used to fabricate the film. According to the compositions provided in Table 1, various weighted amounts of PEG and a fixed amount of PVOH (3g) were dissolved in distilled water and stirred with a magnetic stirrer for 45 minutes at 130°C at 450 rpm to formulate various blends. These chosen

Table 1 — Sample preparation of PVOH/PEG blends

Sample	PVOH (wt%)	PEG (wt%)
PVOH/PEG 10 wt%	90	10
PVOH/PEG 20 wt%	80	20
PVOH/PEG 30 wt%	70	30

compositions of PEG can affect the properties of the synthesized films if different molecular weight of PEG was used to synthesize the composite film based on the previous publications. Thus, synthesized polymer films were denoted as PVOH/PEG 10%, PVOH/PEG 20% and PVOH/PEG 30%.

For the synthesis of natural fiber-based polymer films UCo and TCo of 5 wt% were used in each previously prepared PVOH/PEG blend to make the desired solution. This prepared solution was stirred continuously for 1 hour at 125°C, using a magnetic stirrer until the homogeneous solution was formed. Therefore, the film synthesized with UCo, PVOH and 10 wt% PEG was designated as PVOH/PEG 10%/UCo 5%. Similarly, the film synthesized with TCo, PVOH and 10 wt% PEG film was designated as PVOH/PEG 10%/TCo 5%. The prepared solution was cast on a glass casting plate and allowed to dry completely at standard atmospheric conditions until the composite films were ready to peel off from the casting plates and used for further characterization.

Characterization of Fabricated Films

Characterization Techniques XRD, SEM and FTIR

XRD of all PVOH/PEG, PVOH/PEG/UCo and PVOH/PEG/TCo films was determined with the Rigaku Ultima IV X-ray diffractometer for phase characterization. The patterns were operated at 40 kV and 40 mA. XRD test was conducted at 2°/min and SEM analysis was done with the Quanta 450 model. FTIR was carried out with the Thermo-Nicolet iS5 model. The Fourier-transform infrared spectrophotometer was configured with a resolution of 4 cm⁻¹, covering the spectral range from 500 to 4000 cm⁻¹.

Mechanical Properties

All film samples were cut into the 85 × 15 mm dimensions to evaluate the mechanical properties using an Instron Universal Tensile Machine (UTM) at 2 mm per second speed.

Thickness Measurement

Thickness of the PVOH/PEG, PVOH/PEG/UCo and PVOH/PEG/TCo films was measured using a

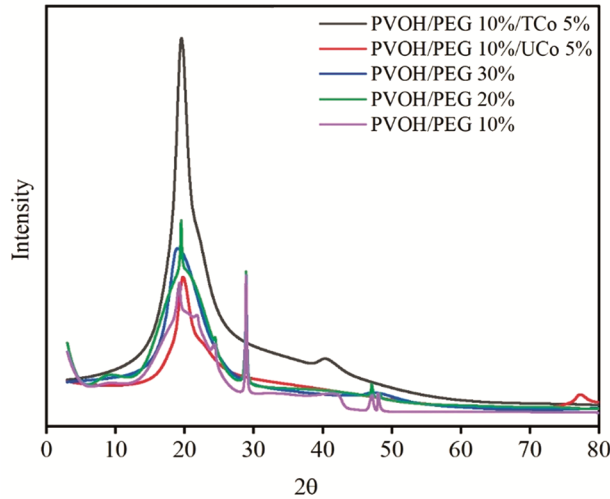


Fig. 1 — XRD pattern of synthesized PVOH/PEG 10%, PVOH/PEG 20%, PVOH/PEG 30%, PVOH/PEG 10%/UCo 5% and PVOH/PEG 10%/TCo 5% films

nickel-plated brass micrometer screw gauge (0–25 mm/0.01mm). Five different points on each of the individual films were marked to evaluate the average thickness.

Swelling Nature

The dried film of each type was cut into 15 × 15 mm size and dipped into a petri dish filled with distilled water at room temperature (35°C) and kept for every 20 sec. Excess water on the surface of film was extracted with filter paper and promptly weighed. The percentage of swelling $S(\%)$ was evaluated as follows:

$$S(\%) = \frac{w_{fo} - w_{io}}{w_{io}} \times 100$$

where, w_{io} is the initial weight of the dry film and w_{fo} is the final weight of the dry film

Results and Discussion

X-Ray Diffraction (XRD)

The patterns created by XRD of all PVOH/PEG films with varied PEG loading and PVOH/PEG/UCo, PVOH/PEG/TCo films are shown in Fig. 1. The peaks at $2\theta = 19.1^\circ$ and at 28.9° revealed by the PVOH/PEG 10% film which signifies crystalline nature due to excellent hydrogen bonding.²⁷ This crystalline nature was decreased as the PEG loading increased²⁸ for PVOH/PEG 20% and PVOH/PEG 30% films depicted by the peaks at 19.5° and 18.9° respectively, due to the distance created between the PVOH

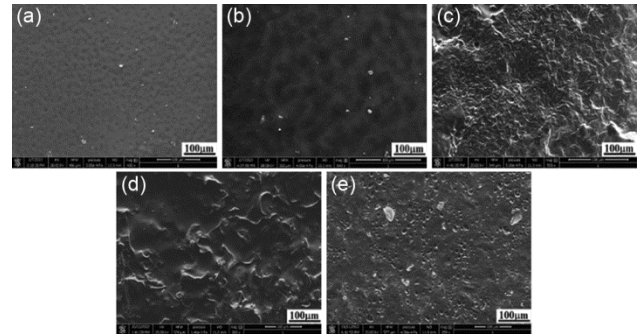


Fig. 2 — SEM micrographs of synthesized films (a) PVOH/PEG 10%, (b) PVOH/PEG 20%, (c) PVOH/PEG 30%, (d) PVOH/PEG 10%/UCo 5%, and (e) PVOH/PEG 10%/TCo 5% films

molecules, caused the decline in a number of hydroxyl groups present per unit volume. Almost similar peaks were observed at $2\theta = 28.9^\circ$ for all PVOH/PEG films, while these peaks were absent in all PVOH/PEG 10%/UCo 5% and PVOH/PEG 10%/TCo 5% films. The untreated coir film (PVOH/PEG 10%/UCo 5%) shows peaks with low intensity at around 19.7° depicting the crystalline behavior of the film and the rest of the region is amorphous.²⁹ In comparison, the high-intensity peak was observed at 19.5° for the PVOH/PEG 10%/TCo 5% film showing an increase in crystallinity due to excellent compatibility among the PVOH, PEG and TCo.³⁰

Scanning Electron Microscopy (SEM)

The morphological analysis of all the films is shown in Fig. 2. A smooth surface was shown by PVOH/PEG 20% film³¹ compared to PVOH/PEG 10% and PVOH/PEG 30% film. Noticeable phase separation was revealed by PVOH/PEG 30% film³² as shown in Fig. 2(c). SEM micrograph of PVOH/PEG 10%/UCo 5% can be seen with smooth and rough surfaces. This surface smoothness might be due to waxy and oily substances present in the untreated coir fiber. Although the treated coir fiber resulted in several pores^{33,34}, better compatibility of the treated coir fiber, PVOH and PEG polymer was revealed, thus increasing the tensile strength of the PVOH/PEG 10%/TCo 5% film.

Fourier-Transform Infrared Spectroscopy (FT-IR)

An FTIR spectrum of PVOH/PEG films, PVOH/PEG/UCo and PVOH/PEG/TCo films are shown in Fig. 3 within the wave number range of $4000\text{--}515\text{ cm}^{-1}$. The spectra of all the films show a significant H-bonding within the wave number range of $3100\text{--}3400\text{ cm}^{-1}$. Hydroxyl functional groups

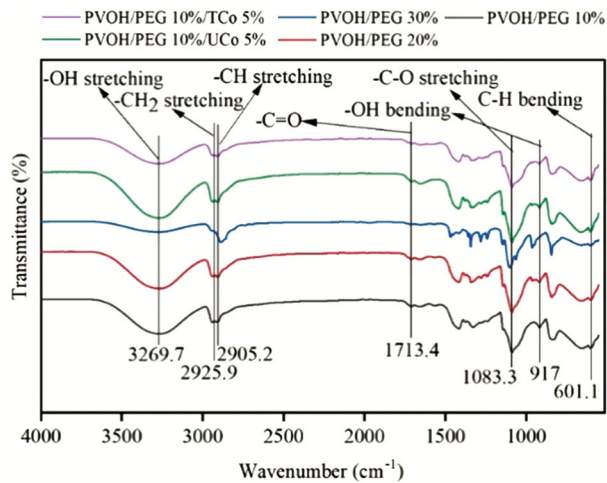


Fig. 3 — FTIR spectra of synthesized PVOH/PEG 10%, PVOH/PEG 20%, PVOH/PEG 30%, PVOH/PEG 10%/UCo 5% and PVOH/PEG 10%/TCo 5% films

exhibit stretching vibrations, which are characterized by³⁵ the wave number at about 3269.7 cm^{-1} . The FTIR patterns of all stretching vibrations of hydroxyl groups ($-\text{OH}$) are found to be similar broad patterns. Although the hydroxyl stretching vibrations pattern of PVOH/PEG, 30% was somewhat flattened due to the immiscibility of PVOH with 30 wt% PEG. The peak at 2925.4 cm^{-1} for PVOH/PEG 10%/UCo5% assigned to $-\text{CH}_2$ stretching was shifted to 2928.3 cm^{-1} for PVOH/PEG 10%/TCo 5% film, while this $-\text{CH}_2$ stretching was absent in PVOH/PEG 30% film. The Peaks at 2905.2 cm^{-1} and 1713.4 cm^{-1} corresponded to $-\text{CH}$ stretching³⁶ and $\text{C} = \text{O}$ stretching vibrations, respectively for PVOH/PEG 10% film were moved to 2900.8 cm^{-1} and 1710 cm^{-1} , respectively for PVOH/PEG 10%/TCo 5% film, while the peak of $\text{C} = \text{O}$ stretching vibrations was absent in PVOH/PEG 30% film and the peak at 1083.3 cm^{-1} associated to $\text{C}-\text{O}$ stretching³⁷ was moved to 1087.1 cm^{-1} for PVOH/PEG 10%/TCo 5% film.³⁸ Hence, this shift of wave number describes the reacting nature of the hydroxyl groups present in the polymers. The 917 cm^{-1} and 601.1 cm^{-1} peaks are correlated with hydroxyl bending and $-\text{CH}$ bending, respectively.

Mechanical Properties

The tensile strength (TNS) and elongation at the break (ELAB) of all the synthesized films are shown in Figs 4–5. The TNS of the PVOH/PEG films decreased while ELAB first reduced and then increased as PEG loading increased from 10 wt% to 30 wt%. However, the addition of untreated coir fiber with PVOH and 10 wt% PEG film can be seen with a

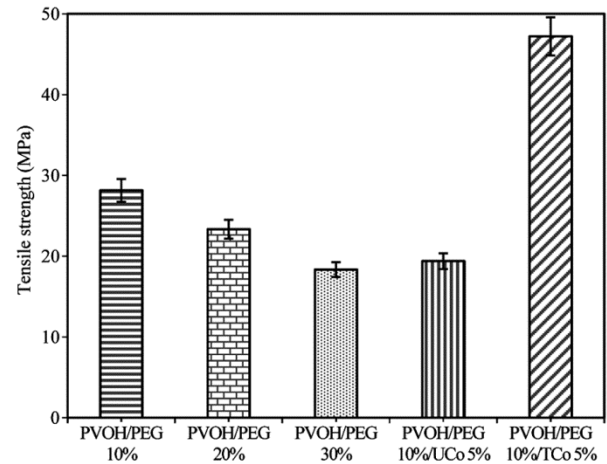


Fig. 4 — Tensile strength (TNS) of the synthesized PVOH/PEG 10%, PVOH/PEG 20%, PVOH/PEG 30%, PVOH/PEG 10%/UCo 5% and PVOH/PEG 10%/TCo 5% films

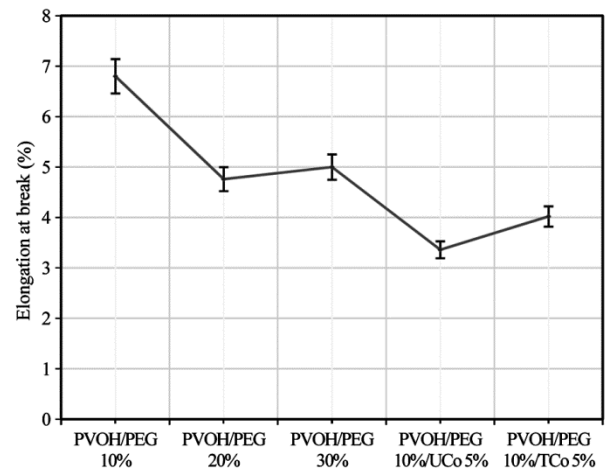


Fig. 5 — Elongation at break (ELAB) of synthesized PVOH/PEG 10%, PVOH/PEG 20%, PVOH/PEG 30%, PVOH/PEG 10%/UCo 5% and PVOH/PEG 10%/TCo 5% films

31.13% decrement in the TNS³⁹ and a 50.58% decline in ELAB⁴⁰ compared to the PVOH/PEG 10% film. In contrast, the addition of alkali-treated coir fiber PVOH/PEG 10%/TCo 5% film resulted in a 67.76% increment in TNS compared to the PVOH/PEG 10% film.⁴¹ The highest TNS of 47.21 MPa was observed for PVOH/PEG 10%/TCo 5% film due to the excellent dispersion of PVOH, PEG and alkali-treated coir fiber. The lowest TNS of 18.33 MPa was observed for the PVOH/PEG 30% film due to poor compatibility of the constituents.

Thickness Measurement

The physical and mechanical properties of the synthesized films are greatly influenced by their average thickness. The average thickness of

Table 2 — Average thickness of the synthesized films

Synthesized film	Average thickness (mm)
PVOH/PEG 10%	0.022
PVOH/PEG 20%	0.027
PVOH/PEG 30%	0.046
PVOH/PEG 10%/UCo 5%	0.152
PVOH/PEG 10%/TCo 5%	0.090

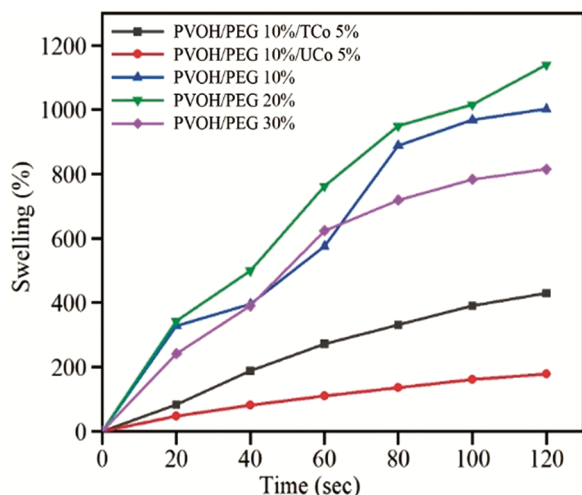


Fig. 6 — Percentage swelling of synthesized PVOH/PEG 10%, PVOH/PEG 20%, PVOH/PEG 30%, PVOH/PEG 10%/UCo 5% and PVOH/PEG 10%/TCo 5% films

PVOH/PEG films increased as PEG content increased, possibly due to the increasing concentration of the solution. The less variation can be seen in PVOH/PEG 10% and PVOH/PEG 20% films due to complete miscibility of polymers. However, a significant variation was observed in PVOH/PEG 30% film than the other PVOH/PEG films as a result of less miscibility of polymers and phase separation. Further, an increase in the thickness was observed with the addition of coir fiber. However, a slight variation in the average thickness can be seen with the untreated and treated coir fiber composite films. The average thickness was evaluated for each film in mm, as shown in Table 2.

Swelling Nature

The percentage swelling of the PVOH/PEG, PVOH/PEG/UCo and PVOH/PEG/TCo films are shown in Fig. 6. The percentage swelling of all PVOH/PEG films was observed to be higher than PVOH/PEG/UCo and PVOH/PEG/TCo films, showing the greater affinity of water molecules for hydroxyl groups present in the polymers.⁴² However lower percentage swelling was seen for PVOH/PEG 30% film compared to other

PVOH/PEG films due to phase separation which was confirmed by the SEM image. The lowest percentage of swelling was observed for PVOH/PEG/UCo film compared to the other films, including treated coir film, due to the excellent reactivity of the constituents. Therefore, the addition of treated coir fiber with the polymers resulted in a decreased percentage of swelling than all PVOH/PEG films. Which is the result of a highly crystalline nature of treated coir film (PVOH/PEG/TCo), confirmed by the XRD test. However, the percentage swelling of PVOH/PEG/TCo film was higher than the untreated coir film (PVOH/PEG/UCo) due to the several factors such as removal of impurities, the opening of the pores and changes of the size of the pores.^{43,44}

Conclusions

In this work, the biodegradable composite film was fabricated by incorporation of PEG with PVOH, treated and untreated coir. It can be concluded that the incorporation of PEG greatly influences the crystallinity of the fabricated film. The SEM micrographs and FTIR spectra revealed the cross-linking of the hydrogen bond among the PVOH, PEG and coir fiber. Further, the incorporation of 30 wt% PEG and above limits its addition with PVOH. During the experiment, the highest swelling percentage showed a significant number of hydroxyl groups present in PVOH/PEG film of 20 wt% PEG loading. Findings from the mechanical properties confirmed that the loading of 10 wt% PEG resulted in the maximum tensile strength for the PVOH/PEG film. Further, the loading of 10 wt% PEG in PVOH/PEG matrix with treated coir fiber resulted in the highest tensile strength compared to all synthesized films. In future, the optimization on preparation of polymers blends of different molecular weight can be employed to synthesize the biocomposite films.

Conflict of Interest

The authors disclose no conflicts of interest.

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