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Characterization of Polyvinyl Alcohol based Biodegradable Film Reinforced with Treated *Imperata cylindrica* (Cogon Grass) Fiber

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ABSTRACT

The increasing environmental concerns regarding plastic pollution have driven the development of biodegradable and sustainable alternatives to conventional synthetic polymers. Natural fibers such as Cogon Grass, when combined with polyvinyl alcohol (PVA), offer promising potential for producing biodegradable films with enhanced properties. In this study, biodegradable films reinforced with Cogon Grass fibers were synthesized using the casting method. Characteristics of the biodegradable film prepared were evaluated by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM). Three analyses were conducted: biodegradation test, water absorption test, and mechanical properties test to study the effect of fiber loading in untreated and treated films. Increased cellulose fiber loading and alkaline treated fiber resulted in a reduction of the intensity of -OH peak in the film. SEM results compared the surface morphology of the control and T-5 film, where heterogeneous surface and void formation were observed. Faster biodegradation was observed in the film with high fiber loading and the film with treated fiber. The lowest water absorption percentage of 142.68 % was observed at the treated film with 5 % concentration of Cogon Grass fiber (T-5). In terms of mechanical properties, significant increase in strength were obtained with fiber loading, reaching a maximum tensile strength of 8.119 MPa for T-5 film. As fiber loading increased, tensile strength and Young's modulus of the film increased while elongation at break decreased. Overall, the findings demonstrated that Cogon Grass could serve as a sustainable reinforcement for PVA-based films, offering a biodegradable material with enhanced strength and environmental benefits.

Keywords: Biodegradable film, Cogon Grass, polyvinyl alcohol (PVA)

1. INTRODUCTION

Millions of tons of waste are generated annually in Malaysia, often containing harmful substances. If this wastes is not managed responsibly, it can cause serious environmental problems. Plastic is one of the largest contributors to waste generation, and it interacts with water and forms hazardous chemicals when dumped in landfills. It then gives harmful effects to the environment and human health (Dana Gopal et al., 2014; Welden, 2020). A single plastic waste takes 100 years for complete decomposition due to its characteristic that consists of high molecular weight and tightly bonded molecules (Bisma Nisar, 2024).

Due to increasing awareness of conventional plastic use towards environment, several studies have been conducted to investigate the substances that can be used to replace non-degradable plastic with the ones that can be degraded. Biodegradable plastic is mostly made from renewable resources, and one of the abundant renewable resources is Cogon Grass. Cogon Grass is a natural fiber that consists of cellulose as an alternative to plastic or film. According to Lokantara et al. (2020), a high content of cellulose in fiber may have good mechanical properties. Despite these advances, there is limited research exploring Cogon Grass specifically as a reinforcement material for polyvinyl alcohol (PVA)-based biodegradable films. Most prior

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studies have focused on other natural fibers or different biopolymer matrices, leaving the potentials of Cogon Grass in film applications. Furthermore, the influence of fiber loading and chemical treatment on the biodegradability, water absorption, and mechanical performance of Cogon Grass–PVA films has not been systematically studied (Loganathan et al., 2022).

According to Desiriana et al. (2015), Cogon Grass contains more than 40 % of cellulose, making it a potential component for making biodegradable plastics, where the process of cellulose bioplastic synthesis consists of two stages. The first step that needs to be done is to isolate alpha-cellulose. The cellulose in the fiber needs to be extracted, which can be done by chemical treatment using alkaline or acid. Alkaline treatment is usually carried out by immersing natural fiber in alkaline at room temperature, or high temperature at a specific period (Ibrahim et al., 2016; Senthilkumar et al., 2018). For acid treatment, the steps include mixing with either dilute or concentrated acid concentrations between 0.2 to 2.5% w/w, and the biomass at a temperature range of 130 to 210°C (Brodeur et al., 2011). However, there are some limitations arising from acid treatment, as it results in inhibitors such as furfural that decrease the effectiveness of this method. The next step is the addition of several additive materials, such as chitosan, glycerol, and oleic acid, to produce three samples of biodegradable plastic. An additive needs to be added in the formation of biodegradable plastic due to cellulose being not resistant to the environment (Desiriana et al., 2015).

This work addresses the problem by synthesizing biodegradable films from Cogon Grass reinforced with PVA using the casting method. It demonstrates the applicability of Cogon Grass fibers as a sustainable reinforcement for PVA-based films, offering new insights into optimizing fiber treatment and loading for enhanced mechanical strength, biodegradability, and reduced water absorption.

2. MATERIAL AND METHODS

2.1 Collection of samples

First, 10 kg of Cogon Grass was collected from abandoned land at Kepala Batas, Pulau Pinang. The Cogon Grass was washed with water to remove all the impurities attached to it. The Cogon Grass was sun-dried until reaching a constant weight to remove residual moisture. Then, the dried Cogon Grass was cut into 2 to 3 cm to form small, fine fibers. The Cogon Grass was ground and sieved with a grinder before screening.

2.2 Preparation of the sample

The treated Cogon Grass was prepared according to the alkaline treatment method proposed by Kommula et al.,(2013). First, 20 g of Cogon Grass powder was treated with 10% w/v sodium hydroxide solution for 2 h at room temperature, maintaining a liquor ratio of 30:1. A 10% w/v sodium hydroxide solution was prepared by dissolving 60 g of sodium hydroxide granules in 600 mL of distilled water. Then, the Cogon Grass was rinsed with distilled water five times before neutralizing it using 1% v/v acetic acid solution, followed by distilled water until pH 7 was reached. The treated Cogon Grass was dried in an oven at 55 °C for 24 h, and kept in an airtight container before blending with PVA.

2.3 Preparation of film

Untreated and treated films were prepared with different fiber loadings of 2.5, 5, 7.5, and 10% of Cogon Grass by the solution casting method. Firstly, a 10% PVA aqueous solution was prepared by mixing 5 g of PVA in 50 mL of distilled water after stirring by a magnetic stirrer for 30 min at 25 °C at 500 rpm. Then, the desired amount of untreated and treated Cogon Grass (2.5, 5, 7.5, and 10%) was added to make a blend with 10% PVA, was stirred again for another 30 min and 10 mL of the solution was poured into a petri dish to produce films. The films were dried in an oven at 55°C for 24 h. The resultant films were named according to the untreated and treated Cogon Grass with different fiber loading, initiating from U-2.5 to T-10, as shown in Table 1.

Table 1: Composition of PVA and Cogon Grass

Samples	PVA (%)	PVA (g)	Cogon Grass (%)	Cogon Grass (g)
PVA (control)	10	5	-	-
U-2.5	10	5	2.5	0.125
U-5	10	5	5	0.250
U-7.5	10	5	7.5	0.375
U-10	10	5	10	0.500
T-2.5	10	5	2.5	0.125
T-5	10	5	5	0.250
T-7.5	10	5	7.5	0.375
T-10	10	5	10	0.500

2.4 Film Characterization

2.4.1 *Functional group analysis*

The FTIR analysis was conducted at 4000-600 cm^{-1}. The film samples produced by different loadings of Cogon Grass were placed into a set holder, and then a suitable spectrum was chosen. The results obtained were presented in the form of a diffractogram of the relation between wave number and intensity, and a spectrometer was used to record the spectrum of FTIR (Fathanah et al., 2018).

2.4.2 Microstructure analysis

The microstructure of the film was characterized using SEM. Each film sample (1 cm x 1 cm) with a different fiber (Cogon Grass) loading was used to investigate the surface structures of the samples. The films were coated first before being put in the SEM equipment. The film samples were placed in the SEM chamber for positioning and image capture at magnifications of 50x and 500x, and a voltage of 15 kV.

2.5 Analysis of Film

2.5.1 *Biodegradability test*

The test was conducted using a soil burial method, where the film was cut into a 2.5 cm x 2.5 cm dimension. Then, the soil was placed inside a box with tiny holes perforated at the bottom of the box. Next, the films were buried beneath the surface soil, which was about 5 cm from the surface. Distilled water was used to moisten the soil surfaces every day. Next, the films were checked and photographed once every 7 days for a duration of 28 days.

2.5.2 Water absorption test

The synthesized film was cut into a dimension of 2 cm x 2 cm before drying in oven at 50°C for 24 hours. After cooling, the film was immediately weighed (W_o). Then, the films were submerged in 50 mL of distilled water for 4 min at room temperature. The films were then taken out from the water and dried using a smooth cloth. The final weight of the films were recorded (W_1). Three samples of each fiber loading were used, and average readings were taken. Absorption of water was measured using Equation 1.

$$Water\ absorption\ (\%) = \frac{W_1 - W_o}{W_o} \times 100 \quad (1)$$

where W_o is the weight (g) of the film sample before immersion, and W_1 is the weight (g) of the film sample after immersion [Ismail & Ishak, 2018].

2.5.3 Mechanical test

The tensile test was conducted at the speed of 5 mm/sec. The films were cut into 40 mm x 10 mm and were placed on the device with the initial distance of 20 mm between grips. The test started when the device pulled the films. The force was shown on the computer screen. The force continued to increase and was then stopped when the film broke (Novianti et al.,2019). Tensile strength (TS) was calculated using Equation 2.

$$TS = \frac{F_{max}}{A} \tag{2}$$

where F_{max} is the maximum load (N) needed to pull the film apart and A is the initial cross-sectional area before test.

Elongation at break (EAB) was calculated using Equation 3.

$$EAB = \frac{I_{max}}{I_o} \times 100 \tag{3}$$

where I_{max} is the film elongation (mm) at the moment of rupture, and I_o is the initial grip length of the film, 20 mm.

Young's modulus (E) was calculated using Equation 4.

$$E = \frac{Stress\ (MPa)}{Strain} \tag{4}$$

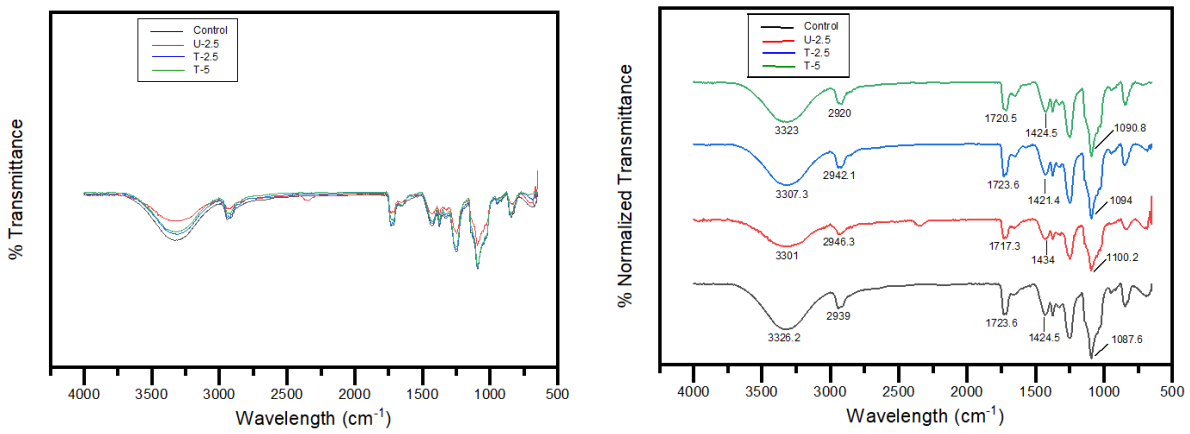
where stress is maximum load (N) divided by initial A (mm^2), and strain is the change in length (mm) divided by the original length (mm).

3. RESULTS AND DISCUSSION

3.1 Functional groups analysis

Figures 1 (a) and (b) display the FTIR spectra of the control, U-2.5, T-2.5, and T-5 films. From the IR spectra of pure PVA that acted as the controlled film, an absorption band at 3326.2 cm^{-1} was observed. This band was assigned to –OH stretching of PVA. The stretching of –OH groups from intermolecular and intramolecular hydrogen bonds among of –OH groups between PVA chains might show high hydrophilic forces (Tan, 2016; Brailson Mansingh et al., 2022). The vibration bands observed at 2939 cm^{-1} was assigned to the C-H stretching from the alkyl group of PVA. The peak at wavenumber 1723.6 cm^{-1} corresponds to C=O stretching of functional group from unhydrolyzed ester (El Achaby et al., 2017), and 1424.5 cm^{-1} corresponds to -OH bending. The bands obtained at 1087.6 cm^{-1} correspond to C-O stretching.

For IR spectrum of U-2.5, T-2.5 and T-.5 films, the peaks observed were similar to the peaks for the controlled film. However, incorporation of cellulose fiber in the PVA caused a slight change of the intensity of –OH stretching. It can be observed that in Figure 1 (a), the intensity of –OH group in U-2.5, T-2.5 and T-5 were significantly reduced as the –OH group of PVA is involved in



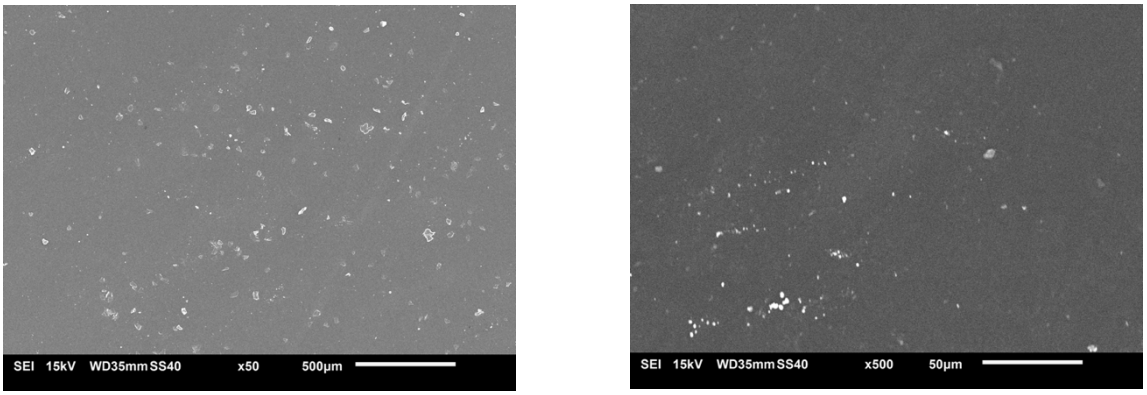
(a) (b)

Figure 1: FTIR spectra of the biodegradable films (a) Transmittance versus wavelength (b) Normalized transmittance versus wavelength

intermolecular hydrogen bonding that increased the polymeric association of –OH group. It can be deduced that hydrogen bonding was formed between the fiber and PVA. Additionally, it was found that the film underwent chemical modification of fibers as it showed some difference in peaks compared to the film with unmodified fiber. It can be observed at absorption peaks of 1094 cm^{-1} and 1100.2 cm^{-1} for T-2.5 and U-2.5. These peaks were assigned to C-O stretching of acetyl group in hemicellulose, where the intensity of C-O stretching in T-2.5 film decreased due to alkaline treatment, were compared to U-2.5 film. Similar decreasing trend in C-O stretching due to the chemical treatment was also reported by Sapuan et al., (2013) as fibers treated by researchers decreased in the lignin and hemicellulose content. Moreover, in treated films (T-2.5 and T-5), treating fiber with alkaline causes the hydrogen bonding in the -OH group to break, thus reducing the –OH groups in alkaline-treated films.

3.2 Morphology analysis

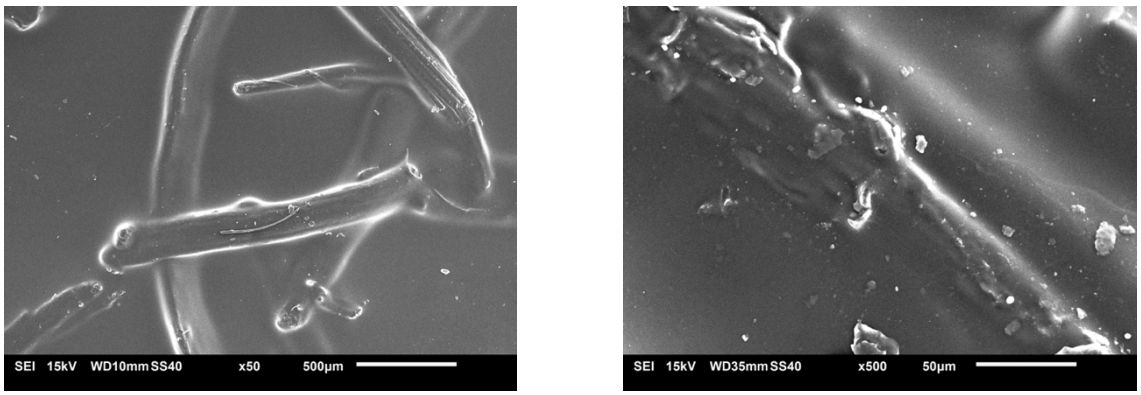
Figure 2 shows SEM images of the controlled and T-5 biodegradable films at 50x and 500x magnifications. It can be observed that the surface of the control film in Figure 2 (a) and (b) was smooth and bubble formation was present.



(a) (b)

Figure 2: SEM images of control film at (a) x50 magnification and (b) x500 magnification

Formation of bubbles was due to high stirring speed of the film solution. For T-5 film as shown in Figure 3 (a) and (b), the cellulose fiber appeared in micro rod-like structures and showed a heterogeneous surface of film. At 500x magnification, a void was observed in the T-5 film. According to Kong et al. (2016), the presence of voids in the film was attributed to bubble formation during the blending of fiber and PVA, which consequently compromised the film's structural integrity.



(a) (b)

Figure 3: SEM images of T-5 film at (a) x50 magnification and (b) x500 magnification

3.3 Biodegradability test

Figure 4 shows the physical appearance of biodegradable films from Cogon Grass fiber subjected to biodegradability tests for 7, 14, 21, and 28 days. The degradation trends indicated biological action on the samples. The control, U-2.5, and U-5 still retained their initial shape while other samples started to degrade after 7 days of soil burial. Increasing the fiber loading in the film caused faster degradation. It was also observed that treated films (T-7.5 and T-10) showed faster degradation than the untreated films at the same fiber loading (U-7.5 and U-10). The control film and U-2.5 showed slight degradation until Day-28, while U-5 shows rapid degradation after 28 days of burial test. This indicates that the biodegradation of the films was strongly influenced by the fiber loading, where higher fiber loading corresponded to a higher film biodegradation in the compost media. The slight degradation in control film may be due to the fact of the C-C backbone linkage of PVA itself, causing low biodegradability (Gulati et al., 2019). On the other hand, Cogon Grass fibers are cellulosic materials that could easily be utilized by microorganisms, causing faster degradation. Thus increasing fiber loadings would increase the amount of microorganism degrading the films. Rapid biodegradation for U-5 film might be due to the high amount of distilled water used to moisten the soil surface. According to Tan (2016), the degradation was also promoted by the surface moisture absorption, that promotes the growth of microorganism. Besides, poor fiber-PVA adhesion may also lead to quickened biodegradation (Rajesh et al., 2015; Syduzzaman et al., 2023).

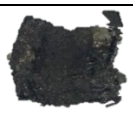
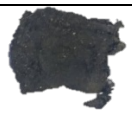
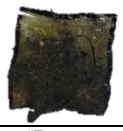
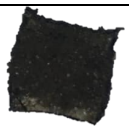
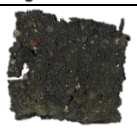
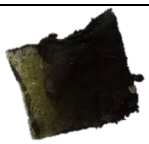
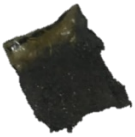
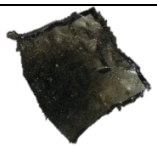
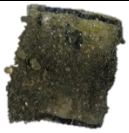
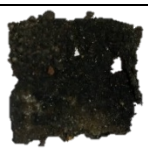
Samples	After burial			
	Day-7	Day-14	Day-21	Day-28
Control				
U-2.5				
U-5				
U-7.5				
U-10				
T-2.5				
T-5				
T-7.5				
T-10				

Figure 4: Effect of fiber loading on biodegradability of untreated and treated Cogon Grass film

3.4 Water absorption test

The effect of addition of different fiber loadings of untreated and treated Cogon Grass fiber in PVA on water absorption behaviour of the films was investigated and presented in Figure 5. In general, all the film samples absorbed water initially and started to dissolve after four minutes of immersion. The controlled film or biodegradable film without Cogon Grass fiber exhibited a much greater water absorption percentage (182.65 %) due to high hydrophilic nature of PVA. The hydroxyl groups (-OH) presence in PVA results in high water absorption, solubility and swelling in water, which is a drawback of PVA film (Jain et al., 2018). The addition of cellulose fiber which

is Cogon Grass to the PVA film composite gave a positive effect on the film as it decreased the hydrophilic nature of PVA as well as decreased water absorption percentage of the film.

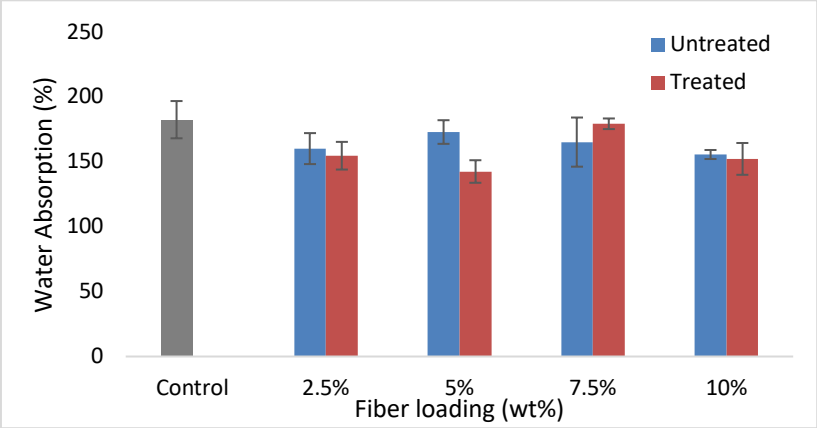


Figure 5: Effect of fiber loading on water absorption of untreated and treated Cogon Grass film

The lowest water absorption percentage of 142.68 % was observed at treated film with 5 % concentration of Cogon Grass fiber (T-5). The fiber content is the main factor influencing the water absorption of the composites. In line with the hypothesis, as the percentage of cellulose fiber increased, the water absorption decreased. The decreased water absorption may be due to the restricted water molecules during penetration, where fiber may serve as the barrier in the film (Ooi et al., 2017). Gulati et al., (2019) also reported increment in cellulose fiber in PVA film resulted in decreased the water absorption. However, water absorption percentage started to increase in U-5, U-7.5, T-7.5, U-10 and T-10 films, which might be due to inadequate mixing (Judawisastra et al., 2017). Super saturation of fiber occurred during the interaction of fiber and PVA leading to an increased free hydroxyl groups, resulting in increased water absorption (Gulati et al., 2019; Loganathan et al., 2022).

It was also observed that water absorption decreased in films containing alkali-treated Cogon Grass fibers compared to films with untreated Cogon Grass fibers. For treated fibers, alkali treatment with 10% w/v sodium hydroxide solution had enhanced interfacial adhesion between natural fiber and matrix, thereby forming a strong bonding between fibers and PVA film. Three untreated films (U-2.5, U-5 and U-10) reported high water absorption of 160.34, 173.12 and 155.79 %, respectively, as compared to the treated films with the same fiber loading (T-2.5, T-5 and T-10). Poor wettability and adhesion between untreated fiber and PVA films might be the reason to this high-water absorption in films (Zakaria et al., 2013; Mohd Hafidz et al., 2022).

3.5 Mechanical test

The mechanical behaviour of biodegradable films was analysed by tensile tests. The comparisons of tensile strength, elongation at break and Young's modulus for different types of biodegradable films with and without treatment of fibers are shown in Figure 6, Figure 7 and Figure 8, respectively. Based on Figure 6, it was observed that all tensile strength of polymer films with addition of fibers in treated and untreated condition increased as compared to the pure PVA film (control). The high tensile strength may due to the hydrophilic nature of PVA which results in strong interface bonding between PVA and hydroxyl groups of fiber (Gulati et al., 2019). Additionally, incorporation of fiber in PVA increased the tensile strength of treated film up to 5 wt% fiber loading and then decreased while for untreated film at 7.5 wt% fiber. Good interfacial

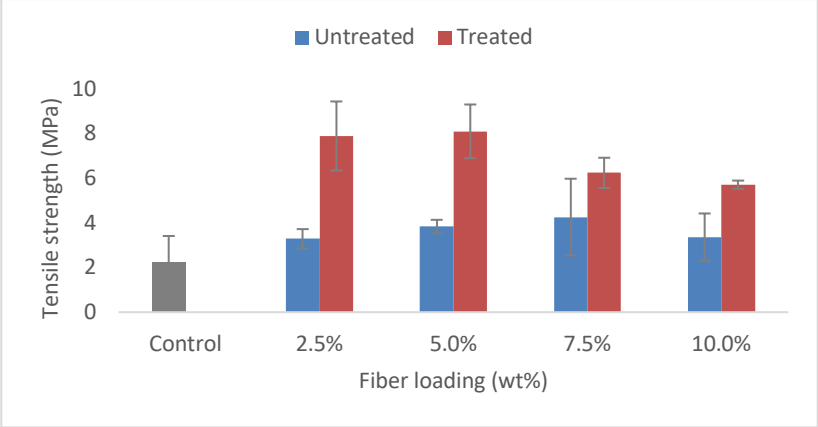


Figure 6: Effect of fiber loading on tensile strength of untreated and treated Cogon Grass film.

adhesion between fiber and PVA matrix may be the reason in increased strength when fiber loading was increased (Syduzzaman et al., 2023). The tensile strength in treated and untreated film drops after reaching the maximum fiber loading, which was 4.267 MPa and 8.119 MPa for U-7.5 and T-5, respectively. Such behaviour may be due to supersaturation of particles in the films as the results of particle-particle interaction instead of particle-PVA interaction (Gulati et al., 2019). Further increase in fiber concentration or loading resulted in increased number of void formations in the film due to poor adhesion between the fiber and matrix. It was observed that the alkali treated fibers improved the film's tensile strength significantly as compared to the ones untreated. The fiber treated with alkali also caused removal of certain hemicellulose and lignin, leading to better mechanical interlocking and better load sharing between fiber and matrix, thus improving the tensile strength.

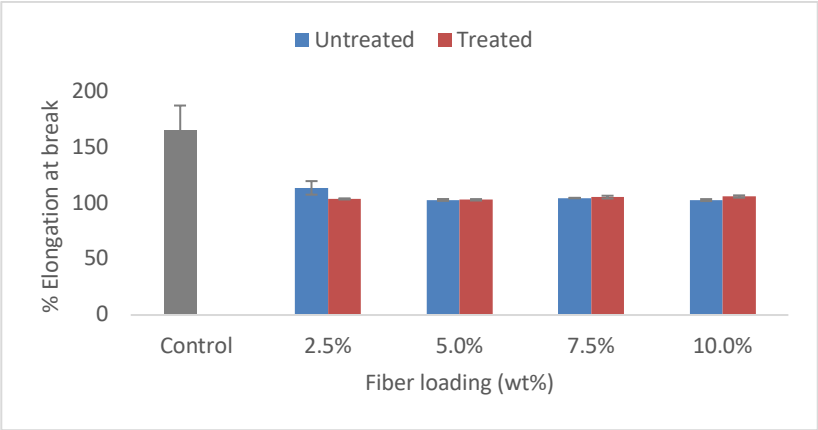


Figure 7: Effect of fiber loading on elongation at break of untreated and treated Cogon Grass films.

Based on Figure 7, it was observed that elongation at break (EAB) of the biodegradable film drastically decreased when Cogon Grass fibers were incorporated into the PVA matrix. The elongation at break decreased with the addition of 2.5 wt% fiber when compared to the control, and stayed unchanged as the fiber content was increased further. Decrease in elongation may be due to high stiffness of the films as the fiber loading increased, the films became more rigid and fiber restricted the stretching of films (Ismail & Ishak, 2018). Thus, the elongation at break reduced as the results of lower deformation behaviour of the films. It was also observed that the treated films exhibited lower elongation at break compared to the untreated films, likely due to a reduction in fiber strength caused by the alkali treatment.

From Figure 8, it can be observed that the Young's modulus (E) of films increased with the increase in the fiber loading up to 5 wt% fiber. As the fiber loading in the film increased, the

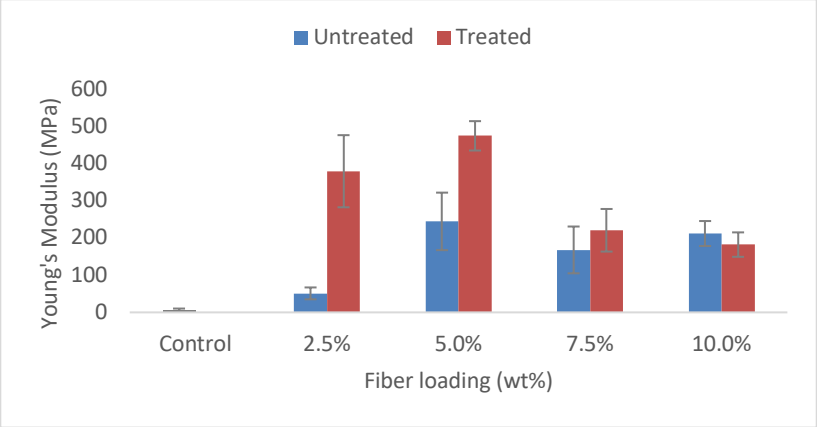


Figure 8: Effect of fiber loading on Young's modulus of untreated and treated Cogon Grass films.

interactions between the fiber and the matrix is improved and crack propagation was inhibited (Laxmeshwar et al.,2012). Decrease in the Young' modulus may be due to the formation of micro cracks in the film due to poor adhesion between the fiber and PVA matrix. It is also observed that, the modulus of the treated films was higher than untreated films. T-5 reported the highest Young's modulus compared to the other films with modulus of 475.58 MPa. This might indicate that chemical treatment of fiber improved the bonding between fiber and matrix by removing natural and artificial impurities.

4. CONCLUSION

Biodegradable film from Cogon Grass were prepared with and without alkali treatment in different fiber loading. The degradation was much faster in films with high amount of fiber and film with alkali-treated fiber. Biodegradable film exhibited a decrease in water absorption with alkali treatment and fiber loading. The tensile strength and Young's modulus of films increased while elongation at break decreased with increased fiber loading and surface modification of fiber with NaOH.

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