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Wettability, Water Absorption, Mechanical and Chemical Properties of Water Spinach Bioplastic

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Starch-based bioplastics is often used in the manufacturing of bioplastics. However, starch-based bioplastic is prone to have low mechanical properties and degradation due to water absorption. Currently, starch-based bioplastics are often added with fiber to increase their resistance to abrasion, mechanical and barrier properties. The addition of water spinach stem, one of the agro-industrial by product with plasticizer and surfactant, will improve the water absorption, mechanical and wetting properties of starch-based bioplastic. This study formulated a bioplastic using water spinach stem (WSS), cassava starch, glycerol, palm oil, and Tween-80 to produce bioplastic films reinforced from a renewable resource. The percentage of WSS was manipulated through the study to observe the water contact angle, water absorption, and mechanical properties of water spinach bioplastic (WSB). The film was casted in the oven at 80 ℃ for 4 hours. Then the WSB was cured at room temperature for 24 hours. The results demonstrated that the highest value of water contact angle was 79.85° at 17 wt% of WSS content. Besides that, 9 and 17 wt% of WSS revealed the lowest water absorption which can maintain for 2 hours. The presence of lignin in WSS enhances the hydrophobicity of WSB. However, greater amount of WSS beyond that would increase the water absorption and hence influence the tensile strength, tensile modulus, and elongation at break of WSB. This alternative opens up possibilities for bioplastics obtained from food waste.

Keywords: Water spinach bioplastic; vegetable-based bioplastic; biodegradable plastics

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The demand for plastic products continues to increase as the use of plastics is essential in our daily life products. These petroleum-based plastics had been produced more than 300 million tons [1]. Subsequently, this plastic waste becomes an environmental threat as it creates high levels of pollution such as greenhouse gas, global warming, and climate change [2]. Therefore, we urgently need an ecological alternative due to the increasing demand of plastic-based packaging film. One of the ecological solution approaches is producing biodegradable plastics or generally called bioplastics. Bioplastics are environmentally friendly relative to petroleum-based plastics, resulting in fewer greenhouse gas emissions such as carbon dioxide, which is one of the main causes of air pollution, global warming, and climate change.

Lignocellulosic fibers, which are made from plant materials, are biodegradable, renewable and widely available, making them a desirable material in order to reduce our dependence on fossil fuels. Currently, food waste or vegetable waste has become of great attention to researchers as sources of lignocellulosic fiber for bioplastic fabrication. Truthfully, 1.3 billion tons of food was wasted worldwide each year including fruits, vegetables, and tubers. Vegetable waste from food industries is commonly pulverized into powder to reduce its weight and ease the disposal process. Vegetable waste can be a significant biomass feedstock in the development of the biorefinery concept [3]. The conversion process in the biorefinery concept includes the concentration and extraction of beneficial elements for nutraceuticals [4], the regeneration of biopolymers [5,6], the anaerobic digestion of waste [7], or the fermentation-based production of biomaterials [8,9]. There were many reports on the production of food-based bioplastics such as avocado [10], corn [11], coconut [12], carrot [13,14], parsley [13,15], radicchio [13], cauliflower [13], cocoa [15], banana [16], jackfruit [17], pea [18], corn [19−21], potato [21], soybean [22], sweet potato [23], rice [10,15,24], spinach [15, 25], and cassava [21,26−31].

Lignocellulosic fibers have many benefits such

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as good reinforcement and filler material, but there are some drawbacks associated with them. These fibers are hydrophilic and have a high degree of crosslinking, which make them incompatible with many biopolymer matrices, that lead to poor interfacial adhesion and reduced mechanical properties [32,33]. Therefore, surface modification to remove the lignin is widely employed but the processes require huge energy input and produce wastewater, which make it technically challenging, expensive and impractical in practice.

Nevertheless, utilizing the fiber as filler incorporated in starch-based bioplastic also generates great benefits, which will create larger surfaces that can increase the mechanical properties, barrier properties and resistance to abrasion. Fiber filler can also propagate specific properties to the final product such as electric conductivity, antimicrobial characteristic, and inherent transparency [34−36].

Therefore, despite many reports on the production of bioplastic from various vegetable waste, there have been limited reports on the water spinach stem (WSS) waste as a filler which is starch-based bioplastic. In addition, water spinach is one of the most consumed vegetables due to its low cost. Therefore, WSS is a common kitchen waste that can be exploited to produce bioplastic. This study explores the development of bioplastic from water spinach (*Ipomoea aquatica*) stem waste and its properties (i.e., physical properties, wettability, water absorption, mechanical, and chemical properties) for food packaging material.

EXPERIMENTAL

Preparation of Water Spinach Bioplastic

Water spinach was collected from a local fresh market located in Shah Alam, Selangor, Malaysia. The leaves were separated from water spinach and the stem was cut into small pieces. The WSS was washed with tap water to remove any dust and soil particles. Then, the WSS was oven-dried at 90 ℃ for 3 h before being grounded into powder. The details of each material used are shown in Table 1. The starch solution was prepared by mixing an aqueous glycerol solution with cassava starch for 30 min at 80 °C using a magnetic stirrer. Then, the WSS powder was added into the mixture at various weight percentages (9, 17, 23, and 29 wt% refer to dry weight cassava starch) and stirred at 80 °C for 5 min. Palm oil and Tween-80 were stirred separately in another beaker and added to the starch mixture. Next, the mixture was stirred at 80 ℃ for another 15 min, poured into a glass petri dish (diameter of 90 mm), and spread into 1 mm thickness. The thin films were heated in the oven at 80 °C for 3 h, then flipped and continued for another 1 h. Finally, the water spinach bioplastic (WSB) film was dried in a desiccator for 24 h.

Morphological Characterization of Water Spinach Bioplastic

Surface morphology and elemental composition were investigated using scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDX) (Thermoscientific Apreo2). About 1 mg of sample was coated with a thin layer of gold and mounted on a copper stub using double-stick carbon tape.

Physical Properties of Water Spinach Bioplastic

The thickness of the film was examined by using a digital micrometer screw gauge (Digimatic Micrometre 293-340, Mitutoyo Corp, Japan). The sensitivity of the film was set to 0.001 mm. The thickness of each dried film was randomly performed at three different places of the films. Next, the weight of the WSB film was measured to determine the density of the WSB film.

Water Contact Angle of Water Spinach Bioplastic

The wettability of WSB was determined using the contact angle method as described by ASTM D5946 [37]. Water contact angle measurements were used to measure the WSB's hydrophobicity. The contact angle was measured using the Image Analysis Workstation and VCA 3000 TM platform. The WSB film with a dimension of 10x10 mm was placed on a contact angle goniometer stage. A water droplet from a syringe containing ±5.0 μL of distilled water was dropped on top of the WSB surface in ambient conditions. The measurement of five different spots on the WSB surface was carried out to determine the average value of the water contact angle for each composition.

Table 1. Weight of materials in water spinach bioplastics (WSB).

Materials	WSB (9 wt%)	WSB (17 wt%)	WSB (23 wt%)	WSB (29 wt%)
Water spinach stem (WSS) (g)	1	2	3	4
Cassava starch (g)	10	10	10	10
Distilled water (ml)	140	140	140	140
Glycerol (ml)	2.5	2.5	2.5	2.5
Palm oil (g)	5	5	5	5
Tween-80 (g)	0.5	0.5	0.5	0.5

Water Absorption of Water Spinach Bioplastic

Water absorption for WSB was determined according to ASTM D570 [38]. The WSB film with a dimension of 20x20 mm was oven-dried at 110°C for 60 min and let cooled to room temperature before the dry weight of the film was weighed. Next, the dried WSB was placed in a closed container for immersion in distilled water for 120 min. The WSB film was removed for each 15 min interval, dried with a cloth, measured the weight, and then returned to the water. These steps were repeated and weighed again after 120 min. The amount of water absorbed was determined by calculating the gain in weight after immersion by the percentage of water absorption from equation (1).

$$\%\ \text{Water absorption} = (\text{Wet weight} - \text{initial dry weight})/(\text{Initial dry weight}) \times 100 \qquad (1)$$

Mechanical Properties of Water Spinach Bioplastic

Tensile strength, tensile modulus, and elongation were measured using an Instron Universal Testing Machine according to ASTM D882 [39]. The initial grip separation was set at 25 mm, and the crosshead speed was 5 mm.min^{-1}. The WSB film was cut into strips with a length of 80 mm and a width of 10 mm. Tensile and elongation tests were replicated five times on each variation of the WSB composition at 23±2 °C and 50±5% relative humidity.

Chemical Interaction of Water Spinach Bioplastic

The intermolecular bonding of WSB was evaluated using an Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectroscopy (Nicolet IS10, ThermoFisher Scientific, USA). The WSB film was first oven-dried at 110°C for 1 h and cooled to room temperature. Next, the dried WSB film was ground to powder before placing the WSB powder to be compressed against the ATR diamond crystal. All spectra were recorded based on an average of 16 scans, a wavenumber range of 650—4000 cm^{-1}, and a spectral resolution of 16 cm^{-1}.

RESULTS AND DISCUSSION

Microstructure and Elemental Analysis of Water Spinach Bioplastic

Figure 1 shows the microstructure of WSB with 9% and 29% of WSS content. WSB with 29% of WSS presented microstructure with great flaws, large pores, and micro cracks (shown by the arrows in Figure 1). On the contrary, WSB with lowest content of WSS presented more continuous surface with more homogeneous distribution of smaller pores. Both samples had the total porosity of 0.189% and 0.290% for WSB film with 9% and 29% of WSS, respectively. It is believed that uneven surface and micro cracks of WSB with 29% of WSS is due to the poor distribution of WSS powder in WSB and increment of agglomeration of WSS powder. Hence, the film is less flexible compared to the film with lower content of WSS. This is due to the insufficient amount of plasticizer in the compound that restrains the flexibility and continuity in the WSB matrix chain [40]. Such uneven surface has also been reported by Ilker et al. [15] in which their surface is densely populated with microcrystallites embedded in the polymeric matrix. It also proved that the agglomeration of the WSS disperses in smaller islands.

Figure 2 shows the EDX spectrum with element mass percentage of the lowest and the highest of WSS content of WSB compositions, which are 9% and 29% of WSS, respectively. The stem of water spinach contained as much as 12% lignin [41] which provided stability to the plant as a whole. Contrary to the starch molecule that has a general formula of $C_6(H_2O)_5$, lignin has a much higher C/O ratio (i.e., chemical formula: $C_{18}H_{13}N_3Na_2O_8S_2$). Hence, a greater proportion of WSS has increased the carbon content in WSB. All the elements such as calcium, magnesium, potassium and phosphorous are also shown in the EDX analysis as reported by Ilker et al. [15].

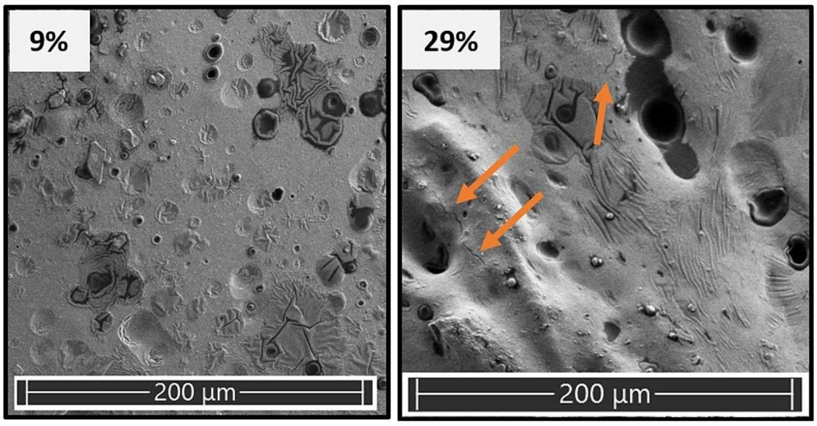

Figure 1. Confocal images of microstructure of WSB with 9% and 29% of WSS.

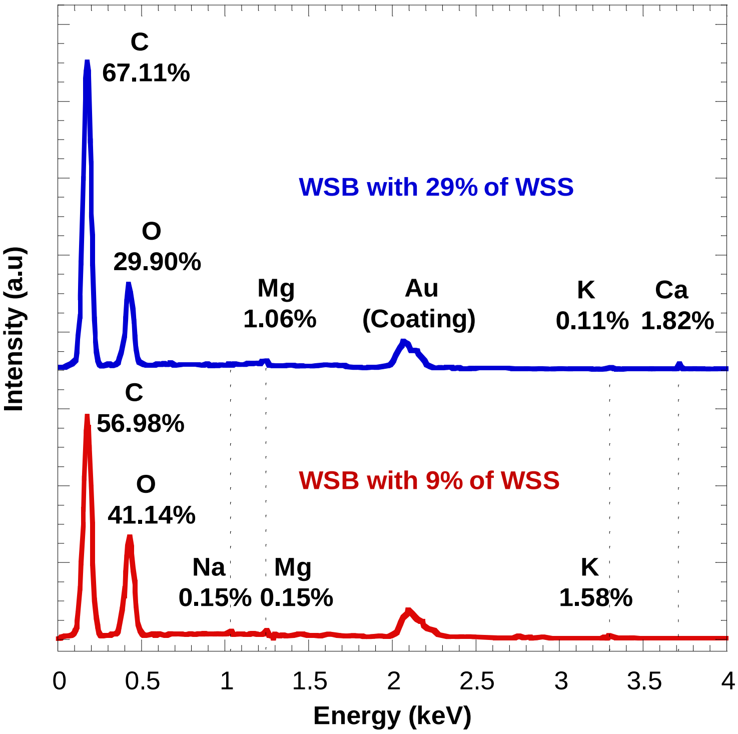

Figure 2. EDX spectrum with element mass percentage of WSB with 9% and 29% of WSS.

Physical Properties of Water Spinach Bioplastic

Figure 3 shows the physical appearance of WSB film. Irrespective of combination of starch, glycerol and WSS, the bioplastic exhibited a yellowish-brown colour. Further increase of WSS in WSB changed the appearance colour from light yellowish-brown to dark yellowish-brown. The WSB films with 23 and 29% of WSS revealed more oily and uneven surface compared to the film with 9 and 17% of WSS. Therefore, it is believed that the increment of WSS content increases the intensity of WSB's colour from light to dark yellowish-brown and influences the distribution consistency of WSS powder in WSB.

Figures 4 and 5 reveal the film thickness and density of various WSB compositions, respectively. The thickness value ranged from 0.863 mm to 1.025 mm.

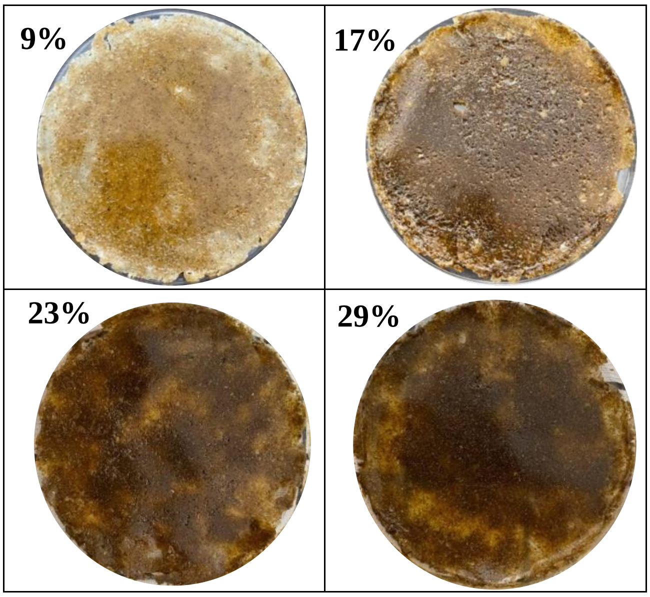

Figure 3. Physical appearance of WSB with different content of WSS.

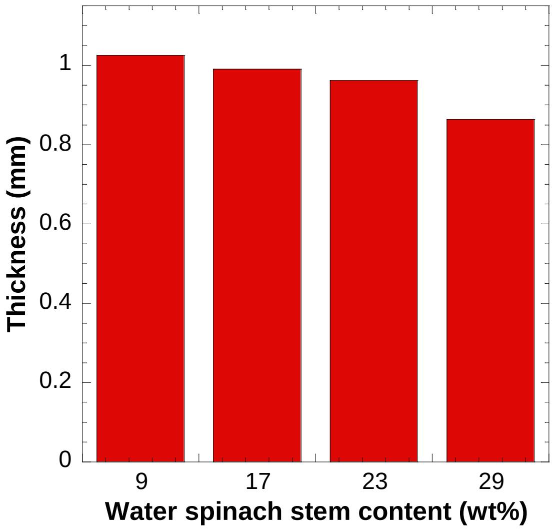

Figure 4. Thickness of WSB with different content of WSS.

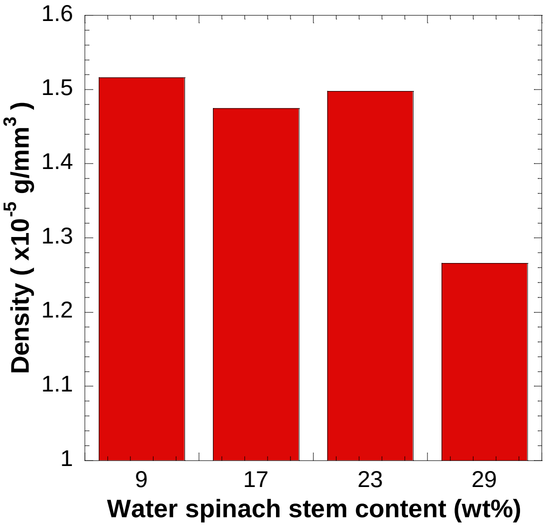

Figure 5. Density of WSB with different content of WSS.

Meanwhile, the density of WSB was in the range of 1.266 to 1.516 x10^{-5} g.mm^{-3}. The WSB with 9% of WSS exhibited the highest thickness and density. This is associated with the influence of glycerol as plasticizer in rearranging intermolecular of polymer structure and transforming all the free volumes in the polymer chain into thicker film which increases the density of the film [42]. However, when increasing the content of WSS, the thickness and density of films are decreased due to insufficient of glycerol in the composition of bioplastic.

Water Contact Angle of Water Spinach Bioplastic

Generally, interaction with water is one of the disadvantages of cellulose-based bioplastics. The interaction of WSB with water is shown in Figure 6. All the WSBs have water contact angles of less than 90º, corresponding to the hydrophilic character. WSB showed a water contact angle of around 80º (17 wt% of WSS), which is similar to vegetable cellulose such as radicchio and cauliflower [13]. The increased contact angle of WSB with 17 % WSS was probably due to the greater proportion of lignin which is a known hydrophobic component of the plant cell wall. Further increase of WSS in WSB decreased the water contact angle. WSS consists primarily of polar lignocellulosic fiber which reasonably acts as a filler in WSB. Increasing the filler will reduce the concentration of plasticizer, which increases the wetting ability and consequently increases the hydrophilicity of WSB [43−46]. The increased hydrophilicity of WSB is also due to the presence of cassava starch and glycerol. A high number of hydroxyl (–OH) groups in amylose and amylopectin of the cassava starch allow polar-

polar interaction with water molecules. Besides, glycerol has a hygroscopic character, which can hold water molecules via absorption or adsorption. Therefore, it increases the hydrophilicity of WSB.

Water Absorption of Water Spinach Bioplastic

Needless to say, water absorption can be reduced by adding additives or using chemically modified starch in bioplastic. The water absorption of WSB is shown in Figure 7. After WSBs were immersed in distilled water for 120 min, the water absorption of WSBs that contained 9 and 17 wt% of WSS were 58.88 and 169.78%, respectively. The other two WSBs that contained 23 and 29% of WSS were partially dissolved in distilled water. The results in Figure 2 reveal a reduction in water absorption (maximum immersed up to 45 min) as the amount of WSS was increased in WSB. However, only low content of WSS can survive in the immersion of more than 75 min.

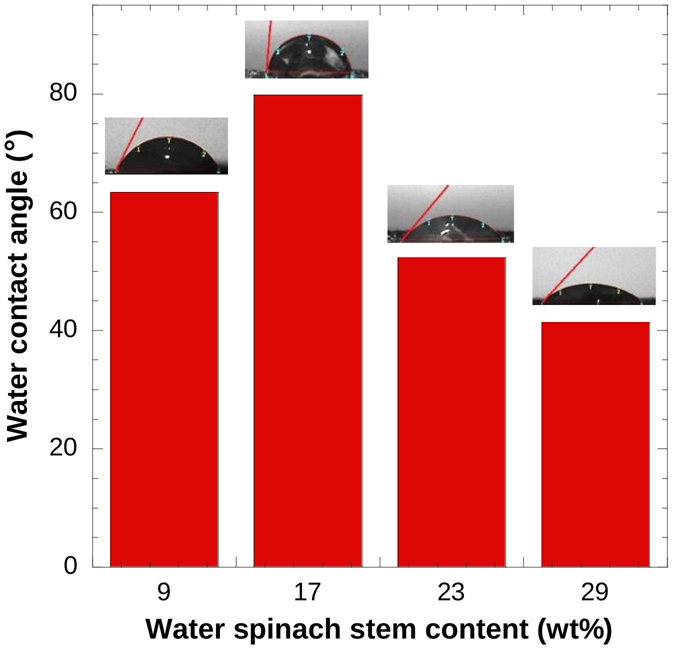

Figure 6. Water contact angle of WSB with different content of WSS.

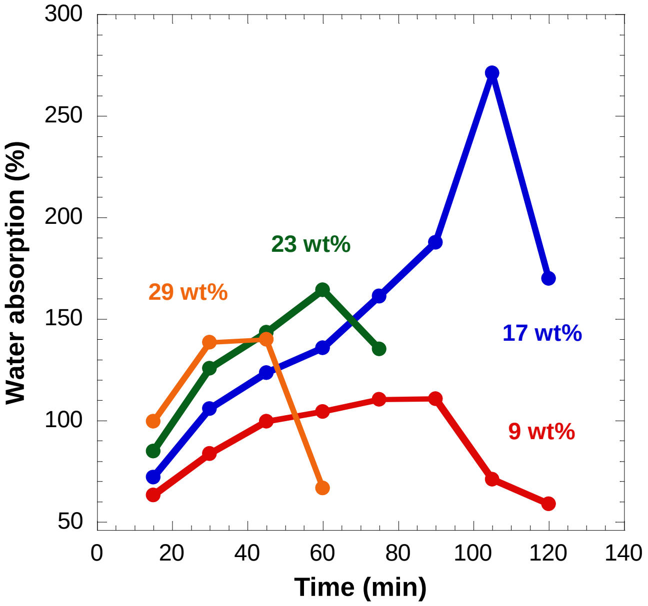

Figure 7. Water absorption of WSB with different content of WSS in 120 min.

This happened due to WSB with a critical concentration of WSS (9 and 17 wt%) allowing the formation of a continuous polymer network with dispersed surfactant for WSS powder to act as a barrier to water [30]. WSS instigates the WSB's resistance to water as the WSS acts as a filler and blocks the pores in the WSB [47]. This pores blockage will probably reduce the interaction of water molecules with WSB and decrease water absorption. A similar reduction of water absorption has been reported due to the addition of filler or fiber in bioplastics [25,36,48].

Table 2 shows water absorption of WSBs containing 9 and 17 wt% of WSS and vegetable-based bioplastics from the literature [18,22,27,40]. The lowest content of WSS in WSB absorbed the lowest amount of water, despite WSB content of 17 wt% of WSS which possessed the highest water contact angle. Furthermore, the WSB with higher content of WSS (23 and 29 wt%) had relatively fast water absorption as it had maximum water absorption and dissolved in water in less than 60 min. The presence of cracks in the film as shown in Figure 1 causes water absorption to be easier and faster. The WSB with higher content of WSS would probably have water that interacts readily with the −OH group in starch. Hydration of −OH groups within WSB starch molecules weakens the hydrogen bond. Therefore, water easily penetrates the film matrix and bonds with more −OH groups, causing the WSB to lose its physical integrity and retention of water weight [28].

Mechanical Properties of Water Spinach Bioplastic

The tensile strength of WSB is shown in Figure 8. The tensile strength of WSB decreased with the increase of WSS content in WSB. Bioplastic with the lowest content of WSS exhibited the highest tensile strength, whereas the highest content of WSS content exhibited the second lowest tensile strength. This is due to the insufficient amount of plasticizer in the compound that restrains the flexibility and continuity in the WSB matrix chain [40]. This is associated with the phase separation between matrix and WSS, poor distribution of WSS powder in WSB, and increased agglomeration of WSS. This similar reduction of tensile strength with the increase of filler was also observed in silica-cassava starch bioplastic [49,50].

Table 2. Water absorption of WSB and various vegetable-based bioplastics.

Materials	Water Absorption (%)	Reference
WSB (9 wt%)	58.88	This study
WSB (17 wt%)	169.78	
Pea protein bioplastic	105.80	[18]
Soybean waste bioplastic	114.17	[22]
Chitosan/sago starch bioplastic	130.31	[40]
Tapioca starch/PVA bioplastic	251.00	[27]
Tapioca starch bioplastic	495.00	

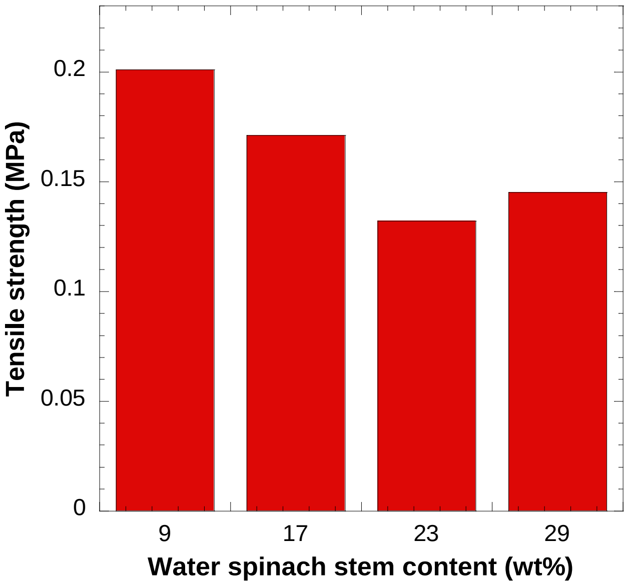

Figure 8. Tensile strength of WSB with different content of WSS.

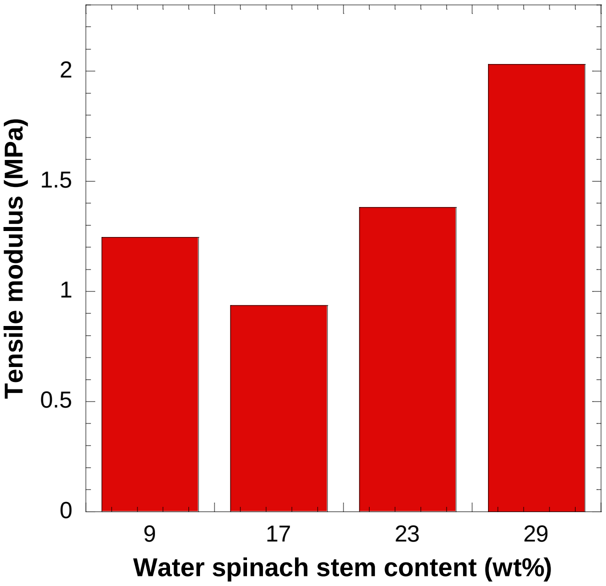

Figure 9. Tensile modulus of WSB with different content of WSS.

The tensile modulus of WSB is shown in Figure 9. It is believed that the higher content of WSS in WSB will increase the tensile modulus due to the increase in the content of crystalline cellulose from the WSS. Similar results was also reported in which the tensile modulus of bioplastic improved as the luffa fiber and kenaf in starch bioplastic were increased [51].

Elongation at break of WSB is shown in Figure 10. The results revealed that the addition of WSS around 9 to 17 wt% will increase their resistance to elongation. However, when the WSS increased more than 23 wt%, the elongation at break decreased due to the decrease of crosslink between the water spinach stem and matrix bioplastic. This is attributed to the insufficient amount of plasticizer in WSB, poor WSS distribution, and the increased amount of agglomeration between the WSS powders. Besides, the plasticizing effect that decreases the free volume of starch molecules also causes the decrease of elongation at break of bioplastic [43].

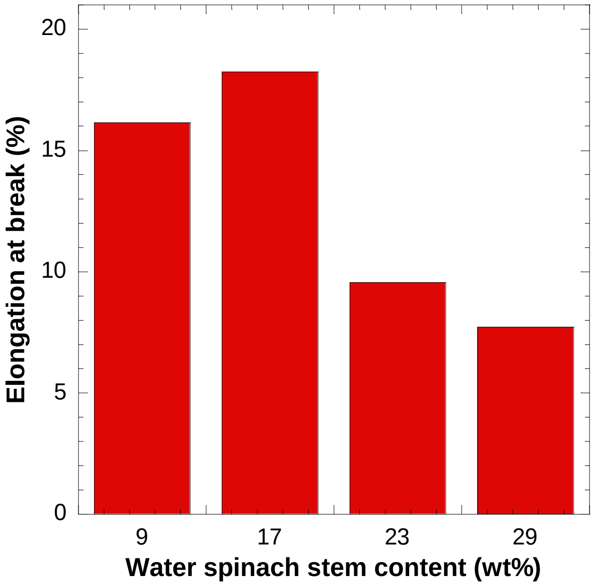

Figure 10. Elongation at break of WSB with different content of WSS.

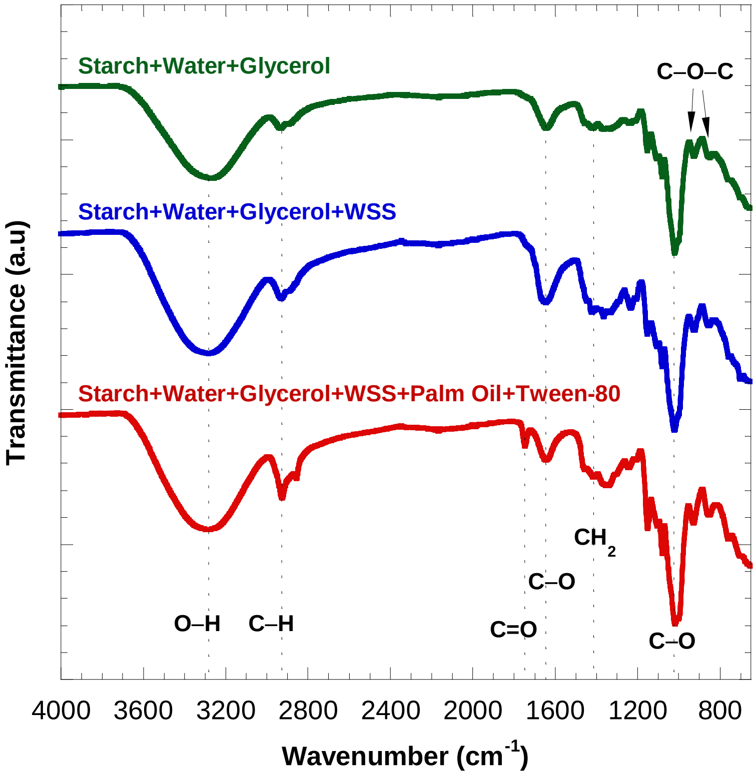

Figure 11. ATR-FTIR spectrum of mixture contained of starch distilled water, glycerol, WSS powder, palm oil and Tween-80.

Chemical Interaction of Water Spinach Bioplastic

Figure 11 shows the ATR-FTIR spectrum of WSB that contained cassava starch, glycerol, WSS powder, and palm oil. The FTIR spectrum revealed the presence of a clear transmittance band attributed to various functional groups of WSB. We can observe the significant variations of bands that are correlated to cassava starch and WSS.

Figure 11 shows the presence of transmittance peaks of cassava starch at 3272, 2924, 1645, 1411, 1016−1077, and 757−926 cm^{-1} which are attributed to O−H stretching, C−H stretching, C−O bending associated with OH group, CH_2 symmetric scissoring, C−O stretching and C−O−C ring vibration of carbohydrate, respectively [21]. The O−H stretching vibration refers to hydroxyl groups and bound water in the cassava starch. It is associated with a decrease in hydrogen bonds between water and the −OH group in the WSB matrix. This peak is more intense with the presence of glycerol in WSB. In addition, peak 2924 cm^{-1} corresponds to the symmetric and asymmetric stretching vibration of the aliphatic C−H group (CH_2) [43]. Furthermore, the transmittance peak at 1745 cm^{-1} is attributed to C=O stretching vibration by the presence of the carbonyl radical in the ester functional group associated with the presence of palm oil. This transmittance peak is also a common presence in the structure of lignin [15].

CONCLUSION

The WSB has been successfully developed using WSS powder as a filler, cassava starch, glycerol, and palm oil. Content of WSS powder in the WSB shows significantly different physico-mechanical properties between various bioplastic film. The rise of WSS content resulted in a significant decrease of tensile strength and elongation at break but concurrently increases tensile modulus. It is worth noting that the intensities of both EDX and FTIR spectrums, and morphological data reflect the results of water contact angle, water absorption and mechanical properties. Incorporation of WSS with glycerol and Tween-80 as a plasticizer and surfactant improves the wettability and water absorption of WSB film at lower content of WSS. The increase of hydrophobicity is probably due to the presence of lignin in WSS. The highest hydrophobicity of WSB is obtained at 17 wt% of WSS with comparable mechanical properties. This could lead to retard the biodegradation of the bioplastic better than bioplastic containing only starch solution. The WSS influences the moderation of the mechanical properties of WSB. Therefore, this research has demonstrated the ability of WSS in the production of bioplastics that could serve as packaging materials, thus protecting and preserving the environment. This can also create a circular economy as one of vegetable waste management.

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