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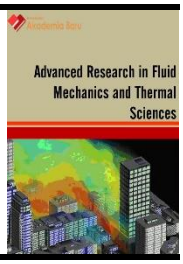

Characteristics of Cogon Grass Fibre Reinforced Thermoplastic Cassava Starch Biocomposite: Water Absorption and Physical Properties

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ABSTRACT

The aim of this study is to investigate the characteristic of thermoplastic cassava starch (TPCS) composites containing cogon grass fiber (CGF) in the range of 1, 3, and 5 wt.%. Initially, the mixture of TPCS and CGF are prepared by pre-mixing using a high-speed mixer at 3000 rpm for 12 min. Then, the thermoplastic composites were pressed at 160°C for 35 min by using a compression molding machine. The composites were characterized for its moisture content, water absorption, thickness swelling and water solubility characteristics. Inclusion of 5 wt.% loading had significantly reduced the thickness swelling and water solubility of the biocomposites by 5.38% and 7.82%, respectively, compared with the neat TPCS. The moisture content and water absorption of the composites did not show significant changes following the addition of CGF. Overall, the incorporation of CGF into TPCS has enhanced the functional properties of the composites for short-life product applications.

1. Introduction

Over the last two decades agriculture-based resources have gained huge attention because of environmental and health concerns, low density, and relatively good performance [1-6]. These agricultural-based resources can be converted into biopolymer and can be used for short shelf-life applications such as container, tray, disposal packaging, etc. [7-9]. Agricultural-based resources is renewable resources that are generally referring to plant based resources such as cellulose, agar and

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starch [10, 11]. Besides, there are also synthetic polymers that are derived from natural monomers or fermentation of microbial which can be considered as renewable materials such as Polylactic acids (PLA), Polyhydroxyalkanoic acids (PHAs), Polyhydroxybutyrate-co-valerate (PHBVs), Polyhydroxybutyrate (PHB), cellulose ester, Polyols as well as plant and animal proteins based [12-14]. Many people have opined that these new bio-based polymers materials have good potential to replace the non-renewable petroleum-based polymers and help solve some of the most crucial pollution caused by the overuse of non-renewable petroleum-based polymer.

Among these biopolymers, starch is considered as one of most promising materials to replace petroleum based polymer due to economic, easy availability, abundant, renewable, biodegradable and requires less or no petrochemicals processes [15-19]. Besides that, the utilization of biodegradable starch biopolymer also was expected to reduce municipal waste and cost operations of landfill. In recent years, thermoplastic starch (TPS) based composite has become the developing environmental friendly product and interesting for packaging applications. According to Jumaidin *et al.,* [20], starch is a heterogeneous materials that consist of two microstructures called amylose and amylopectin. Amylopectin is a highly branched structure of short α-1,4 chains linked by α -1,6 bonds; and amylose is a linear structure of α-1,4 linked glucose unit.

Starch can be transformed into thermoplastic starch (TPS) in the presence of heat, shear, and plasticizer. Under these circumstances, it would result to the formation of homogeneous melt that known as thermoplastic starch. However, TPS has some weakness or limitations in term high water and moisture absorption, high water solubility and low flexibility due to the abundance of hydroxyl groups in their structure. Therefore in order to overcome this drawback issues, many studies have been carried out to improve its properties including blending with other synthetic polymers, chemical modification, graft copolymerization, and by incorporating fillers such as lignin, clay, cellulose, nanocellulose, fibres and multi-walled carbon nanotubes [21-24]. Reinforcing TPS with natural fibre is one of the most promising approaches since it is more cost effective and reliable to the structure. The most common type of starch used for fabricating biopolymer includes corns, wheat, rice, potato, sago and cassava [25].

Cassava also known as *Manihot esculenta* is an agricultural crops species that originates from Euphorbiaceae family. It is ranked the third most widely consumed in tropical regions, and fifth among the starch crop production in the world. Cassava starch has been widely utilized to produce biofilms and the result showed that these carbohydrates are promising materials in this regard [26]. Biofilms that were fabricated from cassava starch biopolymer are described as odorless, colorless, biodegradable, isotropic, tasteless and non-toxic [26]. Renewable and abundant materials such as cassava starch and natural fibre are highly promising candidates for single-used food containers. According to Prachayawarakorn *et al.,* [27] the physical properties of thermoplastic cassava starch (TPCS) was improved when the thermoplastic was reinforced with jute or kapok. This was proved by water absorption test whereby by the addition of cellulosic fibre, the water absorption of TPCS with kapok and jute fiber composites were decreased.

Cogon grass also known as *Imperata cylindrica* is one of the ten most awful weeds in the world because of its ability to effectively colonize, spread, and displace desirable vegetation. This weed is originating from Poaceae family. It is a perennial rhizomatous grass native to east and Southeast Asia, Micronesia, Australia, India, Melanesia and southern and eastern Africa. It can grow up to 3-meter-tall with leave are about 2 cm wide. Currently, cogon grass is used in paper-making, thatching and weaving into bags and mats as well as used for traditional Chine medicine. Cogon grass has been proved to have good weather resistant due to its use as a material for the roof in Thailand [28].

In the present study, cogon grass fibre were used as reinforcement for thermoplastic cassava starch matrix. The aim of this work is to study the effect of cogon grass fibre (as shown in Figure 1) loading on the water absorption, swelling and water solubility properties of the thermoplastic starch. This study reported on the properties of TPCS with glycerol as the plasticizer and cogon grass fiber (1, 3, and 5 wt. %) as the reinforcement. It should be noted that the cogon fibre utilized in this work were not chemically treated and modified. This would lead to the development of low cost materials and production and as well as more environmentally friendly materials. The used of cogon grass fibre as reinforcement agents for TPCS biopolymer matrix added value to this product and increase the TPCS biopolymer composite as totally green food packaging material.

Fig. 1. Cogon grass fibre

2. Materials and Methodology

2.1 Materials

The material cassava starch was acquired from Thye Huat Chan Sdn Bhd (Malaysia). Glycerol used as a plasticizer was purchased from Laboratory chem. The cogon grass was collected from the field near the Universiti Teknikal Malaysia Melaka (Melaka, Malaysia).

2.2 Sample Preparation

The fabrication of TPCS and CGF biopolymer composites is carried out by using TPCS as the matrix and cogon grass fibre as reinforcement of the composites. TPCS is mixed thoroughly where the ratio of cassava starch and glycerol was 100:30 (wt.%). The cogon grass was dried after soaked for a week before the fibre was extracted. The modification of TPCS with cogon grass fiber is performed by incorporating a different amount of cogon grass fibre (1, 3, and 5 wt.%) into the biopolymer matrix. The loading of the reinforcing fibre are shown in Table 1 where the biopolymer matrix was maintained at 100 wt.%. The mixture of TPCS and cogon grass fibre are prepared by pre-mixing using a high-speed mixer at 3000 rpm for 12 min. The resulting mixture is hot-pressed at 160°C for 35 min by using a compression molding machine (Gotech Testing Machine) under the load of 10 tonnes. The TPCS were stored immediately in a desiccator containing silica gel prior to the conditioning process in order to avoid unpredicted moisture absorption.

Table 1
Relative amount of reinforcing materials in composites

Cogon grass fibre (%)	Composites
0	TPCS
1	TPCS/CGF-1
3	TPCS/CGF-3
5	TPCS/CGF-5

2.3 Moisture Content

Five samples were prepared for the moisture content investigation. The samples of thermoplastic cassava starch and cogon grass fiber were heated in an oven for 24h at 105°C. The weight of the samples before (Mi) and after (Mf) heating was obtained in order to calculate the moisture content. The moisture content of the seaweed was determined using Eq. (1).

$$\text{Moisture content (\%)} = \frac{Mi-Mf}{Mi} \times 100 \quad (1)$$

2.4 Water Absorption

The water absorption test of samples is carried out according to the previous study [29]. The samples (10 × 10 × 3 mm) will be dry for 24h at 100°C and cooled in dessicator. Then, the samples directly weighed and then saturated with distilled water at room temperature. The samples will take out from the water after 0.5h and 2h of soaking period. After that, all surface water was softly remove with a dry cloth, and then the samples were weighed again. The differences between the initial weight, Wi and after immersion, Wf of final weights of the samples are calculated by using the Eq. (2).

$$\text{Water absorption (\%)} = \frac{Wi-Wf}{Wi} \times 100 \quad (2)$$

2.5 Thickness Swelling

To remove the existing moisture content from the sample, all five samples (10 × 10 × 3 mm) are being put in an air circulating oven at 105°C ± 2 and dried for 24 h and then immersed in water at room temperature (23 ± 1°C) for 0.5h and 2h. The samples initial thickness, Ti and final thickness, Tf immersion are measured using a digital vernier caliper (Model: Mitutoyo) having 0.01 cm accuracy. The thickness swelling ratio of the laminates for each samples are calculated using the Eq. (3).

$$\text{Thickness swelling (\%)} = \frac{Ti-Tf}{Ti} \times 100\% \quad (3)$$

2.6 Water Solubility

Water solubility (WS) of the samples were determined by measuring method by the permeability of water vapor through the samples with slight modification [16, 30]. From this testing, the samples (10 x 10 x 3 mm) were cut and directly dried at 105°C ± 2 for 24h. The samples were immersed in distilled water with the quantity of 30 ml with gentle stirring after the initial weight of samples (Wo) was measured. The remaining part of the sample was taken from the beaker after 24h of immersion

and the remaining water on the surface was removed using filter paper. Later, to determine the final weight (Wf), the samples were dried again for 24h at 105°C ± 2. The WS of the samples were calculated by the following Eq. (4).

$$\text{Water solubility (\%)} = \frac{Wo - Wf}{Wo} \times 100 \quad (4)$$

3. Results and Discussion

3.1 Moisture Content

Figure 2 shows the moisture content of the composites following the incorporation of CGF. In general, there is no significant changes on the moisture content of the materials with the inclusion of cogon grass fiber into the matrix. The moisture content decrease from 6.4% for TPCS matrix to 5.9% for TPCS/CGF-1 but slightly increase from 5.9% to 7.9% for TPCS/CGF-5. Even though there are small reduction in the moisture content for TPCS/CGF-1, the difference is only 1% which might be due to small variation in the composites. Nevertheless, it is not significant to be taken into account as the effects of fibre. This is consistent with the moisture content results for TPCS/CGF-3 which is almost similar to the neat TPCS. Further increment of CGF for TPCS/CGF-5 shows slight increment which is about 1% of the total moisture content of neat TPCS. Again, this increment is not significant which shows that incorporation of CGF fibre at 1 to 5% has no significant effects on the moisture content of the composites. This might be attributed to the character of CGF which is not relatively hydrophilic than the neat TPCS. Previous study reported that moisture content for carrageenan films increased following the incorporation of grapefruit seed extract into the polymer films, this was attributed due to the hydrophilic character of grapefruit seed than the film matrix [30].

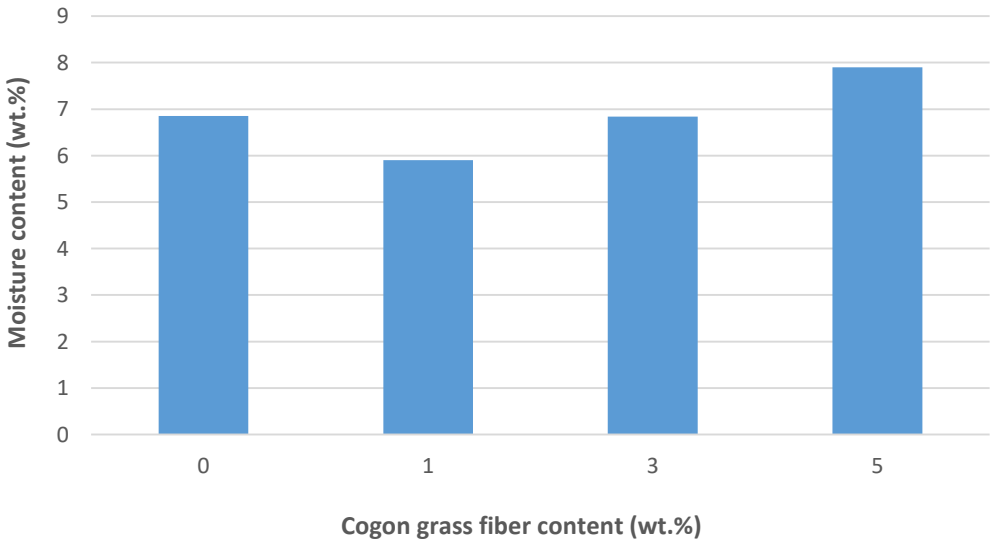

Fig. 2. Moisture content of TPCS with cogon grass fiber composites

3.2 Water Absorption

Biopolymer materials is known to be very subtle to water. Therefore, it is important to investigate the water absorption properties of the totally bio-based material prepared in this study. Water absorption test is commonly used to measure the quantity amount of water absorbed by a material under a particular time. Figure 3 shows the results of water absorption of TPCS/CGF composites with

different fibre loading. In general, the inclusion of cogon grass fiber into the TPCS matrix does not affect the water absorption result and no significant changes can be seen. This result is in agreement with the moisture content results which shows no significant changes in the results. After 0.5h, the results percentage of water contents for all the samples shows increased then slightly decreased, from 48.22 wt.% for TPCS/CGF-1 to 45.23 wt.% for TPCS/CGF-5. Even though there are small increment after the addition of 1% CGF, however, this trend is not consistent for the following composites with higher CGF content. Hence, the fluctuation in the water absorption behavior of the composites might be attributed to the non-homogenous structure of the composites which led to inconsistent water absorption behavior. Again, this finding might be associated with similar characteristic of both fiber and TPCS matrix which led to insignificant changes on the water absorption behavior. After 2 hours, the results show higher water absorption percentage for all composites up to 110 wt.% for TPCS/CGF-1. This might be attributed to longer diffusion process after 2h as well as the hydrophilic properties of the fibers [31]. Again, there is no significant changes on the water content among the composites following the incorporation of CGF after 2h immersion.

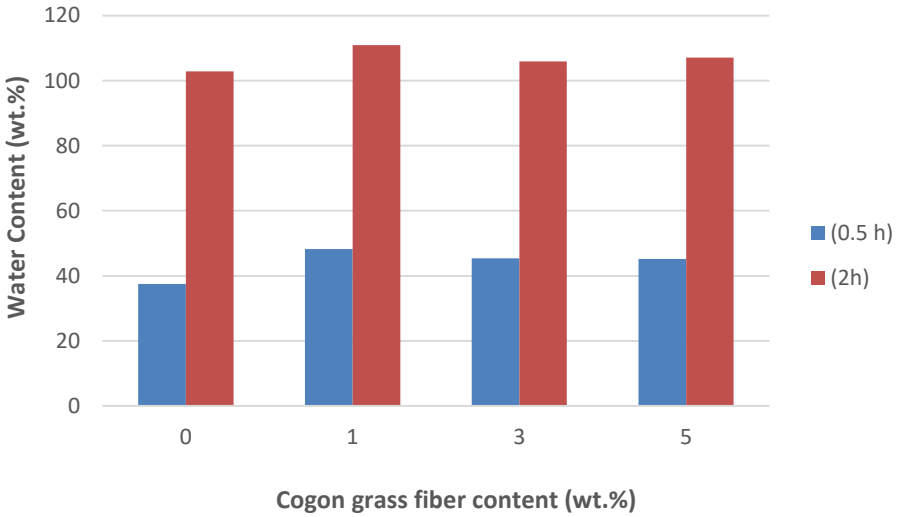

Fig. 3. Water absorption capacity of TPCS/cogon grass fiber composite

3.3 Thickness Swelling

The thickness swelling ratio of TPCS/CGF composites were determined in order to know the changes in dimensional stability of the TPCS following the incorporation of CGF. Figure 4 shows the thickness swelling percentage for TPCS with the inclusion of CGF after immersion for 0.5h and 2h. The thickness of all samples were increased gradually following longer immersion duration from 0.5h to 2h. Similar findings were reported in a previous study for incorporation of sugar palm fiber into thermoplastic starch/agar/seaweed composites [32]. This might be associated with more water molecules being allowed to engage with the hydrogen bonding sites of the composites, which expedites swelling [32]. For the composites, incorporation of CGF led to a lower swelling ratio than the neat TPCS. This effect can be seen after 0.5h of immersion, however it become more significant after 2h immersion. This finding can be ascribed to the presence of fibre in the composites which has more rigid structure than the starch, hence, providing better dimensional stability to the composites. This finding is in good agreement with the previous study on cassava starch/green coir fibers composites [33].

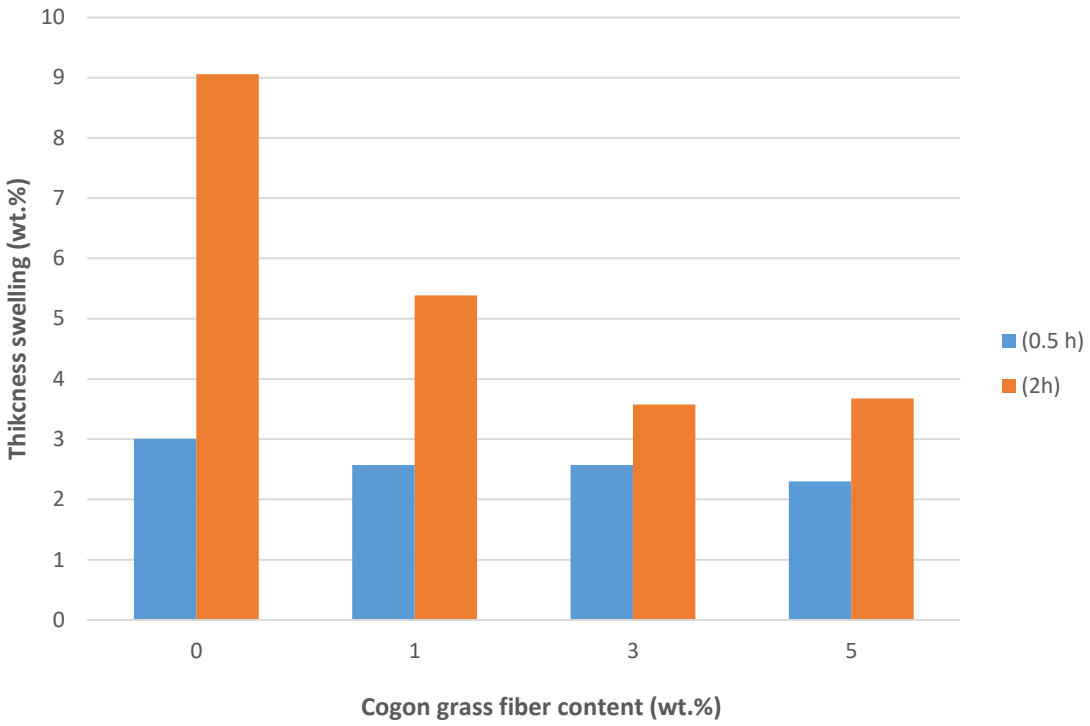

Fig. 4. Thickness swelling capacity of TPCS/cogon grass fiber composite

3.4 Water Solubility

Figure 5 shows the water solubility of TPCS/CGF composites which indicates the water resistance of the materials when subjected to immersion and continuous stirring in water. It was observed that the inclusion of cogon grass fiber into TPCS has reduced the solubility of the composite in the water. The neat TCPS show 36.3% solubility whereas TPCS/CGF-1, TPCS/CGF-3, and TPCS/CGF-5 show 28.70%, 28.90%, and 28.48% solubility respectively. Lower solubility of the composites than the neat TPCS might be attributed to greater water resistance of CGF which aids in hindering water absorption that can lead to disintegration and dissolving of the materials. In addition, the incorporation of natural fibre contributes in preventing disintegration of materials by formation of fibre network in the composites [32]. Moreover, this indicates strong interactions between CGF fibres and TPCS starch chains within the fibre-matrix interface. The ability of CGF in interacting with starch chains might be attributed to the interaction of abundance of hydroxyl groups of CGFs and starch matrix hydrogen bond. Therefore, these interactions provide resistance and stability to TPCS/CGF. Besides, according to Ilyas *et al.,* [34] these interfaces enhanced the cohesive properties of the biopolymer matrix and reduced the sensitivity of water due to water molecules were not able to break these strong bonds. The reduction of water solubility percentage might be ascribed to the greater water resistance of TPCS/CGF composite. Similar results were reported for the addition of hybrid composite from cassava bagasse and sugar palm fiber into TPCS matrix which decreased the water solubility of the films [26]. The authors attributed the findings to the cooperation between the chain of polymer and fibre which reduce the dissolution of the composites.

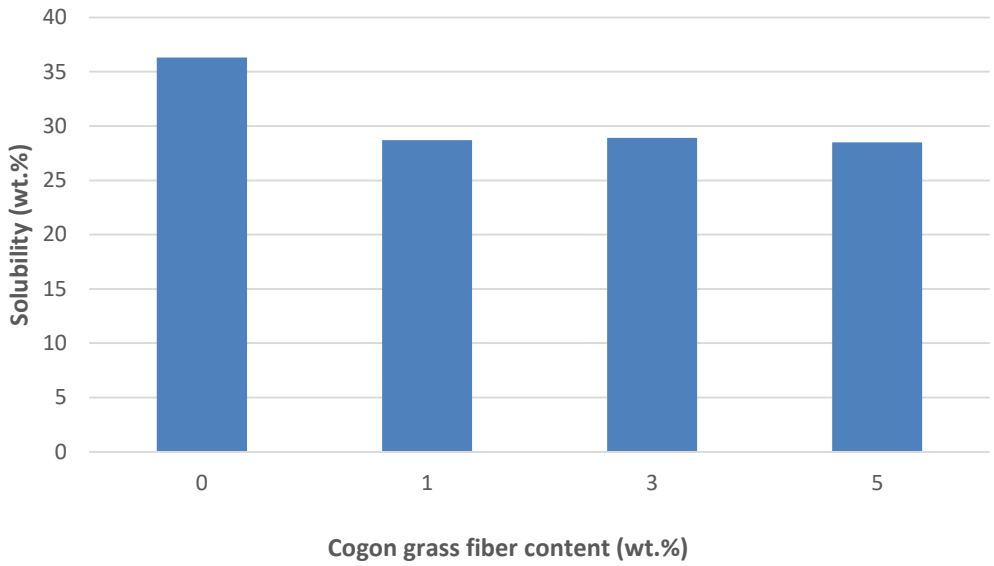

Fig. 5. Solubility of TPCS/cogon grass fiber composite

4. Conclusions

Novel biocomposites derived from TPCS and CGF was successfully prepared via dry mixing and hot pressing. This biocomposites shows variation in the water absorption and physical properties following the reinforcement of CGF. It was found that the thickness swelling and water solubility of the composites decreased with incorporation of CGF which suggested improved dimensional and structure stability of the composites. Meanwhile, there is no significant changes observed on the moisture content and water absorption behavior of the composite. Overall, the composites show improved water barrier properties which is favorable for the potential application as packaging material.

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