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## Cassava/sugar palm fiber reinforced cassava starch hybrid composites: Physical, thermal and structural properties



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#### ABSTRACT

A hybrid composite was prepared from cassava bagasse (CB) and sugar palm fiber (SPF) using casting technique with cassava starch (CS) as matrix and fructose as a plasticizer. Different loadings of SPF (2, 4, 6 and 8% w/w of dry starch) were added to the CS/CB composite film containing 6% CB. The addition of SPF significantly influenced the physical properties. It increased the thickness while decreasing the density, water content, water solubility and water absorption. However, no significant effect was noticed on the thermal properties of the hybrid composite film. The incorporation of SPF increased the relative crystallinity up to 47%, compared to 32% of the CS film. SEM micrographs indicated that the filler was incorporated in the matrix. The film with a higher concentration of SPF (CS-CB/SPF8) showed a more heterogeneous surface. It could be concluded that the incorporation of SPF led to changes in cassava starch film properties, potentially affecting the film performances.

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#### 1. Introduction

In recent years, the use of natural fibers as reinforcing materials in polymers and composites has attracted much attention. The depletion of petroleum resources and the awareness of global environmental problems triggered the search for new green and environment-friendly alternatives [1]. New bio-products derived from renewable plants and agricultural residues, have various advantages, such as low cost, low density, less tool wear, availability, biodegradability and eco-efficiency. Bio-based products enjoys wide applicability in construction, automotive, furniture and packaging industries, which are currently dominated by products based on petroleum feedstock. Therefore, natural fiber is now becoming a new alternative material that can replace petroleum-based products either alone or combined with other materials to produce green composites. The advantages of natural fibers, such as low cost, low density, availability, sustainability, recyclability and biodegradability, make them an interesting research area and considerable efforts are being made to tap their full potential [2,3].

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Cassava is ranked the fifth most widely produced starch crop in the world, and the third among the food sources consumed in tropical regions. The value of harvested cassava can be enhanced by processing cassava to produce cassava starch (CS), thus contributing to the development of rural economies. Among natural polymers, starch has been considered as one of the most promising environmentally friendly materials due to its low cost, easy availability and biodegradability. One of the major by-products of cassava starch is pulp or bagasse. Cassava bagasse (CB) contains 50–60% residual starch (dry weight basis) [4,5]. Biodegradable composite films have been developed from cassava starch/bagasse by casting and dehydration method, using 30% w/w fructose as plasticizer. The results indicated that the addition of 6% bagasse constituted the most efficient reinforcing agent, leading to remarkable physical and mechanical properties of the final product. Although this bio-composite exhibit acceptable properties but still has poor water resistance. In order to overcome this problem, hybridization the CB with lower hydrophilic natural fiber, the natural fiber that known for their high durability and resistance to water is sugar palm fiber.

Sugar palm fibers (SPF) are known for their high durability and resistance to sea water, which are their major advantages. Traditionally, sugar palm fibers were used to make ropes for ship cordages, which were proven to have good properties in sea water.

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The preparation of such fibers is effortless as it does not require any secondary processes, such as water retting or mechanical decorticating to yield the fiber [3,6-8]. In the field of material engineering, SPF has been used as reinforcement in polymer matrix composites. Several studies have shown that sugar palm fibers have great potential to be used in many composite applications like other natural fibers such as kenaf, jute, oil palm, sugarcane bagasse, pineapple leaf and banana pseudo-stem fibers [9–11]. SPF is known to possess water resistance; therefore, the hybridization of SPF with cassava bagasse (CB) seems to be a good combination to improve the properties of the material. The incorporation of two or more different types of fibers into a single matrix is referred to as hybrid composites. The behavior of hybrid composites is a weighed sum of the individual components, in which there is a more favorable balance between the inherent advantages and disadvantages. Also, in a hybrid composite that contains two or more types of fiber, the advantages of one type of fiber could complement what is lacking in the other. Consequently, a balance in cost and performance could be achieved through proper material design [12]. The properties of a hybrid composite mainly depend upon the fiber content of individual fibers, their orientation, extent of fiber intermingling, fiber to matrix bonding and arrangement of both types of fibers. The strength of the hybrid composite is also dependent on the failure strain of individual fibers. Maximum hybrid results are obtained when the fibers are highly strain compatible [13]. Recently, the study of hybrid composites has become an active area in composite material research, the hybrid effect being considered a critical issue in dealing with hybrid composites. The hybrid effect can be positive or negative depending on whether the property in the hybrid composite is greater or smaller than in the non-hybrid composite [14]. However, to the best of our knowledge, there is no information on the preparation and characterization of cassava bagasse/sugar palm fiber reinforced cassava starch hybrid composite films using fructose as plasticizer. The main objective of this work is to investigate the influence of different sugar palm fiber loadings on the physical, thermal and structural properties of the hybrid composite. It should be noted that the fibers used in this study were not chemically treated or modified, which would lead to the development of a more environmentally friendly and cheaper production process and materials. The use of CB and SPF as reinforcement agents for CS thermoplastic film added value to these waste by-products and increase the suitability of CS composite films as environmentally friendly food packaging material.

#### 2. Materials and method

Starch was extracted from native cassava tubers, as described in a previous work [15]. Cassava bagasse was obtained from the same extraction process and was used as filler. Sugar palm fiber was collected at Jempol, Negeri Sembilan, Malaysia. Fructose was supplied by LGC Scientific Sdn. Bhd, Malaysia, and was used as plasticizer.

#### 2.1. Characterization of fibers

#### 2.1.1. Chemical composition

The methods described by (Versino and García, 2014), was used to investigate Acid Detergent Fiber (ADF), Neutral Detergent Fiber (NDF) of cassava bagasse and sugar palm fiber. NDF and ADF were used to determine the Cellulose and Hemicellulose of the fibers. [16]. The proportions of cellulose and hemicelluloses were calculated by using Eqs. (1) and (2), respectively.

$$Cellulose = ADF - lignin$$
(1)

$$Hemicelluloses = NDF - ADF$$
(2)

#### 2.1.2. Water content (WC)

The weight loss was determined to calculate the Moisture content of CB and SPF. Powder samples were weighed ( $W_1$ ), dried at 105 °C for 24 h, and weighted again ( $W_2$ ), WC was calculated as the percentage of initial powder weight lost during drying and that on a wet basis as shown in Eq. (3).

$$WC(\%) = \frac{W1 - W2}{W1} \times 100$$
 (3)

#### 2.1.3. Water absorption (WA)

The water absorption of CB and SPF were determined as per the method explained by Yaich et al. [17]. The samples (3.0g) were dissolved in 25 ml of distilled water and placed in pre-weighed centrifuge tubes. After being kept in the centrifuge for 25 min at 3000 rpm, the dispersions were mixed and were left at room temperature for 1 h. The supernatants were removed and the residue was dehydrated in an oven for 25 min at 50 °C, to determine the moisture content of the samples. The water absorption capacity was denoted as grams of water bound per gram of the sample on a dry basis using Eq. (4), [17].

$$WA(\%) = \frac{M_{\text{final}} - M_{\text{intial}}}{M_{\text{intial}}} \times 100$$
(4)

#### 2.1.4. Thickness swelling (TS)

To determine the percentage of thickness swelling, the samples diameter were measured using a digital micrometer (having 0.01 accuracy) before  $(d_i)$  and after  $(d_f)$  immersed the fibers in water for 24 h, following the method explined by María et al. (2011) [18]. The thickness swelling ratio was calculated using the following Eq. (5):

$$\Gamma S(\%) = \frac{di - df}{df} \times 100$$
(5)

#### 2.1.5. X-ray diffraction (XRD)

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Rigaku D/max 2500 X-ray powder diffractometer (Rigaku, Tokyo, Japan) was used to study X-ray diffraction patterns of the cassava bagasse and sugar palm fiber. The relative crystallinity index (RC) was calculated according to Eq. (6) proposed in a previous work Frost et al. [19], based on the amorphous area (Aa) and crystalline areas ( $A_c$ ).

$$Rc = \frac{Ac}{Ac + Aa} \tag{6}$$

#### 2.2. Film preparation and characterization

Starch films were prepared by casting technique, using a filmforming solution containing 5 g of cassava starch/100 ml distilled water. Fructose was used as plasticizer, in concentrations of 0.30 g/g dry starch. Bagasse was used as filler at 6% w/w of dry starch, the dried fibers were cut and crushed by a Lab grinder and the particle size of cassava bagasse and sugar palm fibers were determined using a 50/100 mesh sieve to obtain powder with particle sizes of 150–300 µm. Sugar palm fiber was added in different concentrations: 2, 4, 6 and 8% w/w of dry starch and were termed CS-CB/SPF2, CS-CB/SPF4, CS-CB/SPF6 and CS-CB/SPF8, respectively. The composition of the films is shown in Table 1. The mixture was heated to 80 °C in a thermal bath and kept at this temperature for 20 min under constant stirring. The air bubbles that formed during heating were removed by placing the film-forming solution into a desiccator under vacuum until there were no more bubbles; then, the solution was poured homogenously onto 10 cm diameter circle plates. The plates with the film-forming solution were then dried in an oven with air circulation, at 45 °C. The dry films were removed from the plates and stored at ambient conditions (around 25 °C and 60% relative humidity) in a plastic bag for two weeks before characterization.

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