



Mechanical and interfacial properties of poly(vinyl chloride) based composites reinforced by cassava stillage residue with different surface treatments

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ABSTRACT

Cassava stillage residue (CSR), a kind of agro-industrial plant fiber, was modified by coupling agent (CA), mechanical activation (MA), and MA-assisted CA (MACA) surface treatments, respectively. The untreated and different surface treated CSRs were used to prepare plant fibers/polymer composites (PFPC) with poly(vinyl chloride) (PVC) as polymer matrix, and the properties of these CSR/PVC composites were compared. Surface treated CSR/PVC composites possessed better mechanical properties, water resistance and dimensional stability compared with the untreated CSR/PVC composite, attributing to the improvement of interfacial properties between CSR and PVC matrix. MACA-treated CSR was the best reinforcement among four types of CSRs (untreated, MA-treated, CA-treated, and MACA-treated CSRs) because MACA treatment led to the significant improvement of dispersion, interfacial adhesion and compatibility between CSR and PVC. MACA treatment could be considered as an effective and green method for enhancing reinforcement efficiency of plant fibers and the properties of PFPC.

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1. Introduction

Plant fibers, the most abundant natural biopolymers on earth, are considered to be an almost inexhaustible source of raw material for the increasing demand of environmentally friendly and biocompatible products [1]. Plant fibers/polymer composites (PFPC), prepared by thermoplastic polymer matrix reinforced with wood or other natural fibers, have received great research interest in recent years because plant fibers have the advantages of low density, low cost, nontoxicity, wide availability, easy processing, high specific mechanical properties, and especially the renewability and biodegradability [2–5]. Currently, the use of agricultural plant fibers for the production of PFPC has drawn much attention as an alternative to traditional wood fibers in plant fibers-reinforced composites [6–9]. Cassava stillage residue (CSR) is the agro-industrial solid residue generated in the still bottoms following fermentation and distillation during the production process of cassava-based bioethanol, and the main component of this residue is plant fiber [10]. A large amount of CSR is generated every year, and most of

this residue has not been efficiently used, or is even burned or discarded as waste, resulting in adverse impact on environment. So, the utilization of CSR as filler material for the production of PFPC can efficiently increase the added value of CSR and avoid environmental pollution [7,10].

Actually, the plant fibers as well as CSR exhibit a high hydrophilicity due to attraction or interaction between the hydroxyl groups of fiber components and water molecules. The interactions between fiber and water originate from the non-crystalline region and extend to the crystalline region [6]. Plant fibers with strong polarity are not well compatible with non-polar and hydrophobic polymer matrix and the interfacial tension between plant fibers and matrix is quite high, leading to weak fiber-matrix interfacial adhesion and poor mechanical properties of the composites [11–13]. In addition, for simple premixed composites, plant fibers have a tendency to form aggregates and separate from the thermoplastic polymers during processing, which provides a poor dispersion of these fibers in matrix and the inhomogeneity of blends, leading to a potential reduction as reinforcement for polymers [9,14,15]. The interfacial adhesion between plant fibers and matrix can be improved by surface modification of fibers, including heat treatment, plasma treatment, corona treatment, alkaline treatment, esterification, etherification, graft

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copolymerization, coupling agent (CA) treatment, and their combination treatments [11,16–22]. Among these methods, CA treatment is considered as a simple and effective method for increasing the interfacial interactions and compatibility between plant fibers and matrix and improving the properties of the composites [23–25]. However, the contact between CA and plant fibers is poor under solid phase conditions because of the highly-ordered and recalcitrant structure of cellulose–hemicellulose–lignin complex and high crystallinity of cellulose in plant fibers [26]. In order to improve the accessibility of plant fibers to CA, it is necessary to adopt assisted means for surface modification of plant fibers.

Mechanical activation (MA), which refers to the use of mechanical actions to change the crystalline structure and physicochemical properties of the solids carried out by high-energy milling, is considered as a simple, effective, and environmentally friendly method for the pretreatment of solid materials attributing to the use of simple and cheap equipment and the operations without the use of solvents, intermediate fusion, etc. [27–29]. The destruction of stable hydrogen bond and crystalline structure induced by MA can efficiently enhance the accessibility and reactivity of plant fibers [30]. Moreover, MA can enhance the dispersion and contact area of plant fibers in thermoplastic matrix due to the size reduction and the split of compact fiber bundles [31]. Herein, we report the MA-assisted CA (MACA) treatment for the surface modification of CSR, and the modified CSR was used as reinforcement for the preparation of PFPC with poly(vinyl chloride) (PVC) as thermoplastic matrix by hot pressing technology. Furthermore, the mechanical and interfacial properties of the PVC composites reinforced with untreated, MA-treated and CA-treated CSRs were also investigated and compared with those of MACA-treated CSR/PVC composite.

2. Materials and methods

2.1. Materials

Cassava stillage residue (CSR) was kindly supplied by Guangxi State Farms Minyang Biochemical Group, INC., China. The cut CSR was comminuted and screened to prepare 40–60 mesh size (0.25–0.38 mm) particles. The comminuted CSR was then oven-dried at 105 °C for 4 h (moisture content < 3%) before use. The dried CSR was uniformly mixed with 5% w/w calcium carbonate (weight percent compared to CSR) that had been treated by 30% w/w stearic acid (weight percent compared to calcium carbonate) to improve the flowability of CSR, and this mixed CSR was designated as untreated CSR. PVC was obtained from Nanning Chemical Co., Ltd., China, and was used as polymer matrix. Aluminate coupling agent (ACA, DL-411-A) was purchased from Nanjing Unite Chemical Co., Ltd., China. All other chemical reagents were of analytical grade without further purification and were obtained commercially.

2.2. Surface treatments of CSR

The MACA surface treatment of CSR was carried out in a customized stirring ball mill driven by a commercially available drill press equipped with a speed-tuned motor [32]. A fixed amount of milling balls (300 mL, 6 mm diameter) was first added into a jacketed stainless steel chamber (1200 mL), and then 40.0 g of CSR, 0.4 g of ACA were added into the chamber. The mixture was subjected to milling at the speed of 375 rpm at a constant temperature of 80 °C. The balls were removed after the sample was milled for 25 min, then the chamber and milling balls were thoroughly cleaned and dried before the next milling experiment. The resulting sample was oven-dried at 105 °C for 2 h and then sealed for standby.

MA treatment of CSR was operated the same way as in MACA treatment, except for the non-addition of ACA.

CA treatment of CSR was performed in a GF-1100 high-speed mixer (Laizhou Huarun Chemical Machinery Factory, China). The CSR was first added to the high-speed mixer and stirred while gradually increasing the temperature. When the temperature reached 80 °C, 1% w/w ACA (weight percent compared to CSR) was added and the modification of CSR was carried out for 25 min under high-speed stirring at the speed of 1000 rpm. The CA-treated CSR was oven dried at 105 °C for 2 h and then sealed for standby.

2.3. Preparation of CSR/PVC composites

For each composite preparation experiment, PVC and 1% w/w Ca-Zn composite heat stabilizer (weight percent compared to the total weight of CSR and PVC) were first added in the GF-1100 high-speed mixer to mix uniformly, and then the CSR sample (50 wt.%, weight percent compared to the total weight of CSR and PVC) was added in the mixer. After thorough mixing, the compound was removed from the mixer. The resulting mixture was compression-molded in an XLB25-D plate vulcanizing press (Huzhou Xingli Corp., China) to produce the composite specimens in a preheated press at 190 °C under a pressure of 6 MPa for 12 min.

2.4. Mechanical properties of the composites

The stress–strain properties of the composites were measured by an Instron DNS-100 universal testing machine (Changchun Testing Machine Research Institute, China). The flexural strength was measured at a crosshead speed of 2.0 mm/min according to ASTM D790, and the tensile strength was measured at a crosshead speed of 5.0 mm/min according to ASTM D638 [33]. All these tests were performed at room temperature.

2.5. Scanning electron microscopy (SEM) analysis

Morphologies of fracture surfaces of the composite specimens were performed using an S-3400N scanning electron microscope (Hitachi, Japan). The samples were fixed on a sample bench using double-sided tape, and then a thin layer of gold was coated on the samples prior to measurement to improve the conductivity. SEM micrographs with different magnifications were obtained to observe the surface morphologies of different composites.

2.6. Water immersion test

Water absorption of the composites was determined following ASTM D570 [11]. The dried composite specimens were immersed in deionized water and maintained at 20 °C for 24 h. The water absorption (W_a) was represented as follows:

$$W_a = \frac{(W_t - W_0)}{W_0} \times 100\% \quad (1)$$

where W_0 is the initial weight of dried composite specimens and W_t is the wet weight of the composites immersed in water for 24 h.

The dimensional changes of the composites were measured by comparing the length, width and height of each specimen before and after water immersion. The changes for length (L_c), width (W_c) and height (H_c) were given by:

$$L_c = \frac{L_t - L_0}{L_0} \times 100\% \quad (3)$$

$$W_c = \frac{W_t - W_0}{W_0} \times 100\% \quad (4)$$

$$H_c = \frac{H_t - H_0}{H_0} \times 100\% \quad (5)$$

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