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The Effect of Fumigation and Alkalization on Dimensional Changes of Cantala Fiber Reinforced Unsaturated Polyester Composites Under Immersion

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Keywords:

Cantala fiber
Dimensional changes
Water immersion
Fumigation treated
Alkali treated

Abstract

This study investigates the effect of fumigation and alkaline treatment on dimensional change in cantala fibers reinforced Unsaturated Polyester Resins (UPRs) composite under immersion test in an aqueous environment. Composites were fabricated using the compression molding method with cantala fiber content of 30% vt with the addition of 5% microcrystalline cellulose. Composite specimens were immersed under aquades for two months at room temperature. The dimensional changes of cantala fiber-reinforced UPRs in different solutions followed a Fickian behavior. The dimensional change of composites in solution shows the saturation point after several weeks of immersion. Fumigation-treated fibers and alkali-treated fiber composites after immersion have lower results in dimensional changes and slope than untreated fiber composites.

1 Introduction

In the industrial field, the materials needed are high-quality materials such as high mechanical properties by their usefulness, competitive material and manufacturing prices, and ease of recycling materials [1]. One alternative that can meet the material criteria is to use biocomposites. Biocomposite is a composite whose constituents consist of at least one natural material, either fiber or matric [2]. Biocomposites have a tremendous impact on the environment because they can be renewed. In addition, natural fibers have good mechanical properties, low density, require low energy during production, and are easy to recycle [3]. Cantala fiber is one of the natural fibers developed in Indonesia. One of these agave plant species has intense, complex, and capable of growing in dry weather [4].

Unsaturated polyester has high mechanical strength supported by low price and is easy to fabricate. It has been widely applied to automotive and construction [5]. One of the problems in the manufacture of natural fiber composites is the lack of distribution of natural fiber cellulose. One of the efforts to overcome that is by reducing particles of natural fiber cellulose compounds to increase the spread in the composite using Microcrystalline Cellulose (MCC) [6]. MCC is pure cellulose isolated from α -cellulose using mineral acids with the advantages of renewable, inexpensive, non-toxins, high mechanical properties, and biocompatibility [7].

<https://dx.doi.org/10.20961/mekanika.v21i1.49463>

Revised 17 March 2022; received in revised version 18 March 2022; Accepted 21 March 2022
Available Online 31 March 2022

2579-3144

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In polymer composites, fiber amplifiers have mechanical properties influenced by the adhesion bond between fibers and matrices. Fumigation is a method that aims to make the moisture content of fibers decrease. It causes the surface of the fiber to become rough, resulting in the composite's mechanical properties increasing [8]. Previous research on the alkaline treatment of natural fibers to the tensile strength of composites obtained from alkaline treatment can increase the bonds between fibers and matrices by eliminating lignin and hemicellulose in natural fibers [9].

Previous research explained that the exposure of natural fiber/thermoset composites under room temperature immersion significantly decreased the mechanical properties [10]. The water immersion might directly affect the dimensional [11]. To complement the analysis of immersion behavior, the research about the dimensional change of cantala fibers reinforced UPRs composite in aqueous environments needs to be done. The tests were conducted to complete the research analysis: thickness dimension change and surface micrograph observation.

2 Experimental Methods

2.1 Materials

Unsaturated polyester has a specific gravity of 1.04-1.46, tenacity value of 1-5%, and tensile strength value of 89.63 MPa, which is the highest value compared to other thermoset resins [12]. Cantala fiber is a natural fiber derived from the extraction of the leaves of the plant *Agave Cantala Roxb.* *Agave Cantala Roxb.* plant is widely grown in Kulonprogo, DIY to Temanggung, Central Java. Based on the Research and Industrial Development of the Ministry of Industry, Yogyakarta cantala fiber has a cellulose content of about 64.23%, so it has the potential as a composite reinforcing fiber [13]. The content of cantala fiber consists of 64.23% cellulose, moisture 13.13%, ash 4.98%, lignin 5.91%, and extractive 1.1%. Microcrystalline Cellulose (MCC) is pure cellulose derived from α -cellulose. MCC is obtained from various natural sources, such as woody plants, cotton bark, straw, and cane pulp isolated by delignification method using the alkaline solution, bleaching, and α -cellulose hydrolysis using a high-temperature acid solution [15].

2.2 Fiber treatment

Cantala, a fiber fumigation process, is one of the chemical treatments to affect surface roughness in a composite. The fumigation process is carried out for 10 hours using smoke from burning coconut fibers. The temperature used in the fumigation process is between 40 °C to 60 °C to produce much smoke and not to heat the cantala fibers. The alkali treatment was applied by immersing cantala fiber in a NaOH solution for six hours. After treatment, cantala fiber was washed in a 1% CH₃COOH solution to remove any traces.

2.3 Fabrication

Composite manufacturing process using compression molding method with the pressure of 10 MPa with a duration of 12 hours and room temperature. Then, the composite is inserted into the oven for a two-hour post-cure process with a temperature of 60 °C.

2.4 Water immersion

The specimens were immersed in distilled water (aquades). The dimensional changes were recorded by periodically removing the specimen from the water bath and using an electronic balance of 10⁻⁴ g to monitor the mass during the aging process. The samples were weighed until the weight of water content reached equilibrium. The dimensional change graphs were calculated from its thickness before, h_0 , and after, h_t as follows (see Equation 1).

$$h_{max} = 100\% \times \left(\frac{h_t - h_0}{h_0} \right) \quad (1)$$

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The slope of dimensional change (S) was calculated from the difference of thickness versus the square root of time by (see Equation 2).

$$S = \frac{h_t - h_0}{t_t - t_0} \quad (2)$$

3 Results and Discussion

Observation of dimension changes in immersion testing in this study using a percentage of dimension change (h_{max}) shown in Equation 1. The curve of dimensional change in immersion test in aquades water is shown in Figure 1. The curve of dimensional change results from the average percentage of dimension change (h_{max}) of all specimens. At the start of the observations, dimensional changes in the solution increased rapidly until, after a few weeks, it reached the saturation point. The phenomenon indicates that the curve of dimensional change follows Fickian behavior, where the dimensional change of composites in solution shows the saturation point after several weeks of immersion [14].

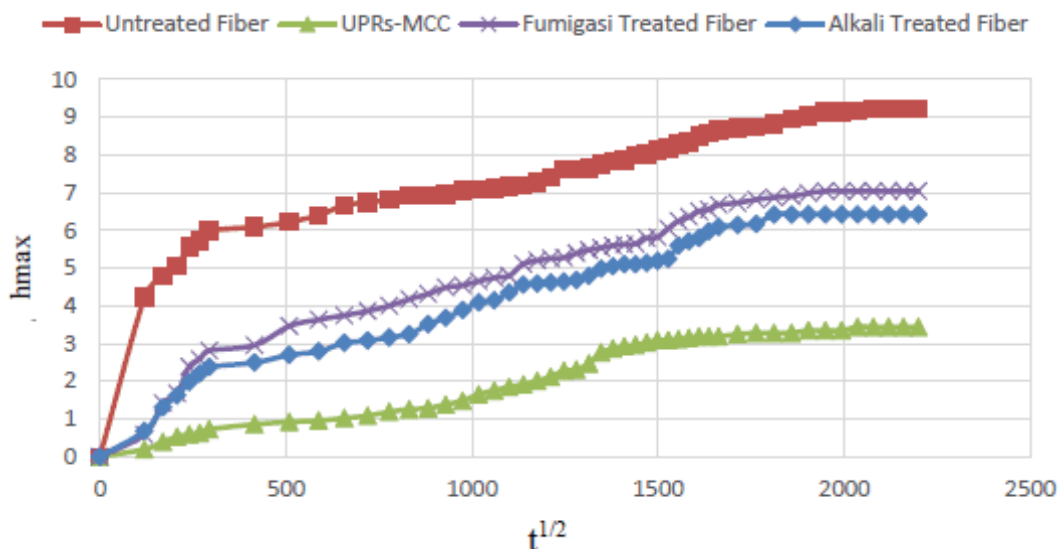


Figure 1. The curve of dimensional change of composites in a solution of aquades

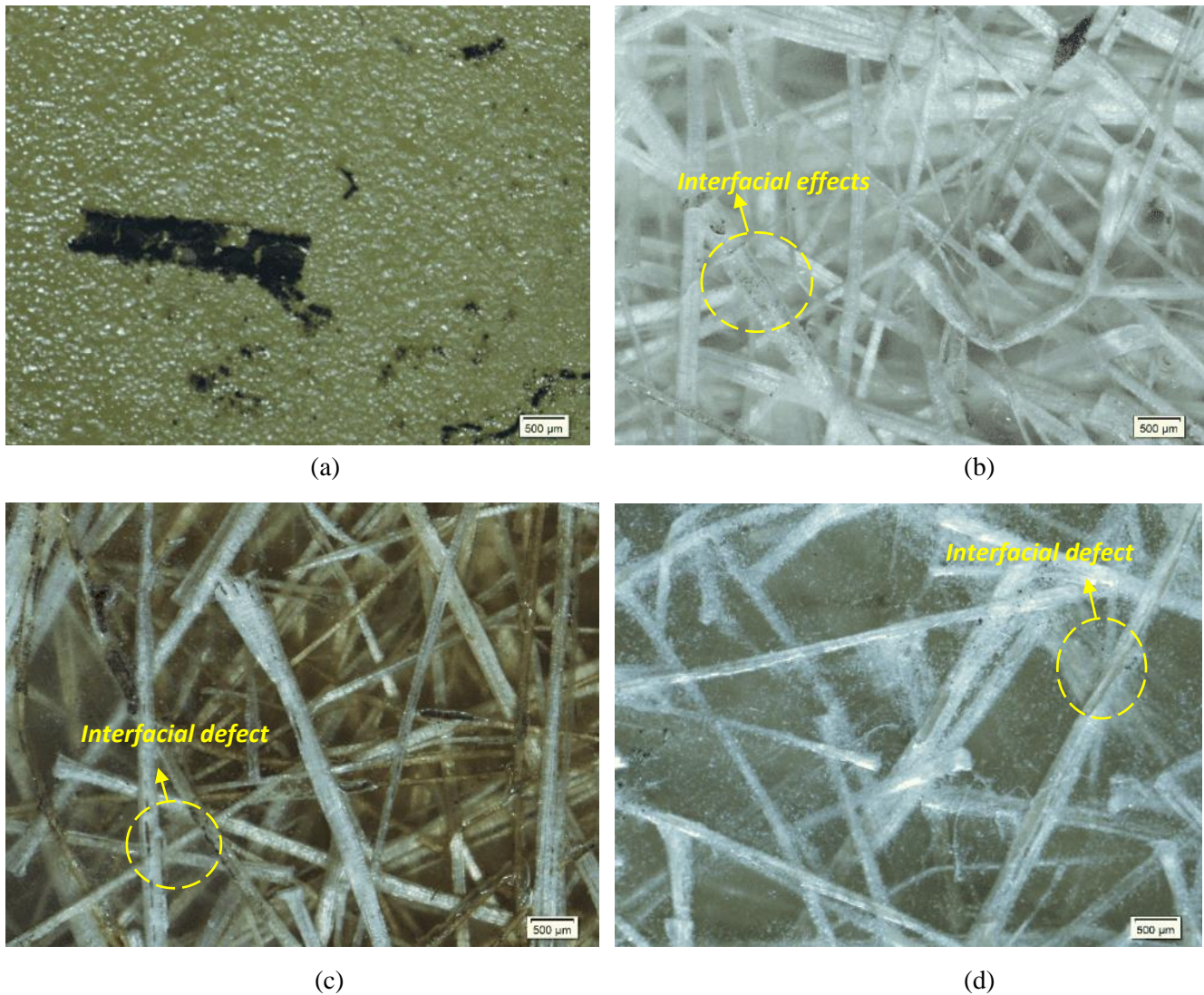
In specimens of untreated cantala fiber composite shown in Figure 1, there are the most significant water absorption phenomena in aquades fluid. This phenomenon occurs because the result of fumigation and alkaline treatment of fibers affects water absorption. A previous study by Ariawan et al. explained that the alkaline treatment of fibers reduced the water absorption rate associated with the elimination of hemicellulose and lignin. Hemicellulose absorbs water more quickly compared to cellulose crystals. In specimens with fumigation, fiber treatment also applies the phenomenon because fumigation treatment can reduce the content of hemicellulose in fibers [15,16].

The slope of dimensional change (S) and maximum dimensional change (h_{max}) in Table 1 are sequentially each type of specimen untreated cantala fiber composite > specimens of fumigation treated cantala fiber composite > specimens of alkaline treated cantala fiber composite > UPRs-MCC specimens. In Table 1, the value of the slope of dimensional change (S) and maximum dimensional change (h_{max}) is directly proportional to the greater the value of slope of dimensional change (S), the greater the value of maximum dimensional change (h_{max}). The phenomenon is influenced by immersion that lasts for 60 days, leading to reduced strength of adhesion bonds between fibers and matrix. It also causes microscopic cracks or voids in the composite.

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Table 1. The slope of dimensional change (S) and maximum dimensional change (h_{max}) in aquades

Fluid Type	Aquades			
	UPRs-MCC	Untreated Fiber	Fumigasi Treated Fiber	Alkali Treated Fiber
S (%/h)	0.008	0.018	0.013	0.013
h_{max} (%)	4.239	9.212	7.218	7.091

**Figure 2.** Macro surface image of the specimen after immersion on aquades: (a) UPRs-MCC, (b) Untreated cantala fiber composite, (c) Alkali treated cantala fiber composite, and (d) Fumigation treated cantala fiber composite

Phenomena occurring in Figure 2 indicate the onset of gaps between fibers and matrices that tend to be more numerous. Figures 2 (b), 2 (c), and 2 (d) show that slits and cracks tend to be more due to swelling of the fibers occurring on the composite surface of the cantala-fiber UPRs without fiber treatment, with fumigation fiber treatment, and with alkaline fiber treatment after immersion when compared to UPRs-MCC specimens. This phenomenon is supported by Najafi et al. The amount of fiber used as a matrix reinforcement is directly proportional to the mixed ability to water absorption [17]. Cracks that occur in specimens due to the disconnection of interface bonds between fibers and matrices that start from fiber bending and the phenomenon that also occurs in fiber damage.

4 Conclusions

Cantala fiber-reinforced unsaturated polyester composites were successfully created using process compression molding. Specimens with 30% vt of cantala fiber content have revealed that dimensional change patterns followed Fickian behavior. The slope of dimensional change (S) and maximum dimensional change (h_{max}) on untreated cantala fiber composite are higher than fumigation cantala fiber composite. Alkaline treated cantala fiber composite and UPRs-MCC. In addition, fumigation and alkaline treatments in cantala fiber slightly reduce changes in composite dimensions. So that phenomenon can be concluded that composite ups-fiber cantala without treatment absorbs the most fluid compared to other composite specimens in aquades with a value of $S = 0.018\%/h$ and $h_{max} = 9.212\%$. The phenomenon is supported by the observation of macro surface images on specimens.

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