Journal of Natural Fibers

ISSN: (Print) (Online) Journal homepage: www.tandfonline.com/journals/wjnf20

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To cite this article: Chin-Hao Yeh, Jyh-Horng Wu & Teng-Chun Yang (2024) Drying Shrinkage and Mechanical Strength of Cementitious Composites with Alkali-Treated Makino Bamboo Fibers, Journal of Natural Fibers, 21:1, 2321531, DOI: 10.1080/15440478.2024.2321531

To link to this article: https://doi.org/10.1080/15440478.2024.2321531

Published online: 06 Mar 2024.

JOURNAL OF NATURAL FIBERS
2024, VOL. 21, NO. 1, 2321531
https://doi.org/10.1080/15440478.2024.2321531

Drying Shrinkage and Mechanical Strength of Cementitious Composites with Alkali-Treated Makino Bamboo Fibers

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ABSTRACT

Alkali-treated bamboo fibers (AFs) treated with different NaOH concentrations (5 and 10%) and treatment times (1, 12, and 24 h) were used as fillers to fabricate cementitious composites with AFs (CAFs) in the present study. The results demonstrated that alkali treatment caused the partial decomposition of hemicellulose and lignin and an increase in the surface roughness of bamboo fibers. Additionally, the amounts of calcium hydroxide in alkali-treated CAFs were higher than in untreated CAFs, and they increased with increasing NaOH concentration and treatment time. For drying shrinkage (DS) under 75% relative humidity (RH), the DS values of the CAFs significantly decreased after adding AFs compared to the DS values of untreated CAFs. Compared to untreated CAFs, the density of the 5% and 10% NaOH-treated CAFs with longer treatment times decreased by 2.9% and 5.1%, respectively. Furthermore, the tensile strength of all alkali-treated CAFs exhibited no significant differences when compared with that of untreated CAFs, while the modulus of rupture and compressive strength were significantly decreased by NaOH treatment. These results indicated that the AFs significantly improved the drying shrinkage of the CAFs and hydration retardation effect of cement pastes, while the density and mechanical strength of the CAFs decreased.

摘要

本研究采用不同NaOH浓度（5%和10%）和处理时间（1、12和24小时）的碱处理竹纤维（AF）作为填料，制备了含有AF的水泥基复合材料. 结果表明，碱处理使竹纤维的半纤维素和木质素部分分解，表面粗糙度增加. 此外，碱处理的CAFs中的氢氧化钙含量高于未处理的CAF，并且随着NaOH浓度和处理时间的增加而增加. 对于75%相对湿度（RH）下的干燥收缩率（DS），与未处理的CAFs相比，添加AF后的CAFs的DS值显著降低. 与未处理的CAFs相比，处理时间较长的5%和10%NaOH处理的CAF的密度分别降低了2.9%和5.1%. 此外，与未处理的CAFs相比，所有碱处理的CAF的拉伸强度没有显著差异，而NaOH处理显著降低了断裂模量和抗压强度. 这些结果表明，AF显著改善了CAFs的干燥收缩和水泥浆体的水化阻滞效果，同时降低了CAFs的密度和机械强度.

KEYWORDS

Alkali treatment; bamboo fiber; cement; drying shrinkage; makino bamboo; mechanical strength

关键词

碱处理; 竹纤维; 水泥; 干燥收缩; makino竹; 机械强度

Introduction

Sustainable cement-based materials have been developed by adding natural fibers to fabricate green cementitious composites in concrete technology. Since natural fibers are biodegradable, cost-effective, and readily available worldwide, they are used as environmentally friendly fillers to replace conventional synthetic fibers and steel rebars in cement paste. Many studies have shown that natural fibers as reinforcement improve the mechanical properties and energy absorption capability of cement matrices

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(Awwad et al. 2012; Yan and Chouw 2014; Yan, Chouw, and Jayaraman 2014; Lecompte et al. 2015; Yan, Su, and Chouw 2015, an et al. 2016; Onuaguluchi and Banthia 2016; Li, Zhou, and Guo 2017; Banjo Akinyemi, Temidayo Omoniyi, and Onuzulike 2020; Sanchez-Echeverri, Medina-Perilla, and Ganjian 2020; Z. Zhang and Angst 2022). Among these natural fibers, since bamboo fiber (BF) has rapid renewability and high specific mechanical properties, several researchers have investigated the use of BFs as fillers in cementitious composites (Akinyemi and Omoniyi 2018; Banjo Akinyemi, Temidayo Omoniyi, and Onuzulike 2020; Coutts, Ni, and Tobias 1994; Li, Zhou, and Guo 2017; Sanchez-Echeverri, Medina-Perilla, and Ganjian 2020; Sudin and Swamy 2006). These results showed that BFs could reduce the weight of the cementitious composite and significantly enhance the mechanical strength of cement. Moreover, bamboo, like other natural fibers, is a lignocellulosic material and has a hydrophilic nature, resulting in drawbacks for the cementitious composite, such as reduced workability of cement paste (Islam, Hussain, and Morshed 2012), hydration retardation of cement (Bishop and Barron 2006; Chakraborty, Kundu, Roy, Adhikari, et al. 2013; Jo and Chakraborty 2015), fiber degradation in an alkali environment (da Costa Correia et al. 2019; Savastano et al. 2009), lower bonding behavior between fibers and cement matrix (Banjo Akinyemi, Temidayo Omoniyi, and Onuzulike 2020; Chakraborty, Kundu, Roy, Chakraborty et al. 2013; Guo, Sun, and Satyavolu 2020; Li, Zhou, and Guo 2017; Sanchez-Echeverri, Medina-Perilla, and Ganjian 2020; Yan et al. 2016), and higher shrinkage of cement (Bederina et al. 2012; Guo, Sun, and Satyavolu 2020; Mohr, Biernacki, and Kurtis 2006; Roma, Martello, and Savastano 2008; X. Zhang et al. 2021). Therefore, the type and characteristics of natural fibers are one of the factors affecting the properties of cementitious composites with natural fibers.

Among the various modification approaches for lignocellulosic fibers, alkali treatment (i.e., with sodium hydroxide, NaOH) as an important chemical method has been used to improve the hydration retardation effect of cement, the mechanical properties of the cementitious composite, and the compatibility between fiber/cement interfaces (Banjo Akinyemi, Temidayo Omoniyi, and Onuzulike 2020; Jo and Chakraborty 2015; Li, Zhou, and Guo 2017; Sanchez-Echeverri, Medina-Perilla, and Ganjian 2020; Yan et al. 2016; Yang, Hua, and Hu 2023; Yin and Li 2022). Yan et al. (2016) indicated that 5% NaOH (20°C for 30 min)-treated coir fibers improved the compressive properties and flexural properties of cementitious composites compared with untreated composites due to the improvement in fiber/cement interfacial adhesion. Li et al. (2017) investigated that the mechanical strength of oil well cement supplemented with increasing amounts of 10% NaOH (23°C for 24 h)-treated BF from 0 to .7 wt%, but there was an accompanying reduction in compressive properties. Sanchez-Echeverri et al. (2020) reported that adding 5% calcium hydroxide-modified BFs increased the flexural strength of the cement by approximately 40% compared to the composite with untreated BFs. As mentioned by Jo and Chakraborty (2015), the findings indicated that alkali-treated (5% NaOH for 24 h) jute fibers increased hydration reaction products in cementitious composites compared to those in composites with untreated jute fibers. Additionally, better bonding between fibers and cement matrix in the alkali-treated composite was observed from the SEM image due to an increase in the surface roughness and the fibrillation of the jute fibers after alkali treatment. Yin and Li (2022) reported that the cementitious composite with 3% NaOH-treated bamboo fibers showed the better mechanical properties and met their domestic product specifications. Yang et al. (2023) stated that 1 wt% bamboo fibers, which were disintegrated after alkali treatment, improved drying shrinkage and slightly increased in the specific tensile energy of cementitious composites. As described above, most studies have primarily focused on the effects of fiber content on the properties of cement with untreated and alkali-treated fibers (Banjo Akinyemi, Temidayo Omoniyi, and Onuzulike 2020; Jo and Chakraborty 2015; Li, Zhou, and Guo 2017; Sanchez-Echeverri, Medina-Perilla, and Ganjian 2020; Yan et al. 2016; Yang, Hua, and Hu 2023). To date, little work has been reported on the drying shrinkage and mechanical strength of cementitious composites with bamboo fibers that were treated with various NaOH concentrations and treatment times. In Taiwan, makino bamboo (*Phyllostachys makinoi*) is an economically important bamboo. Additionally, makino bamboo has higher mechanical properties than other bamboo species (Chung and Wang 2017; Yang and Lee 2018; Yang, Chung, and Yeh 2023; Yang, Hua, and Hu 2023; Yeh and Yang 2020). Therefore, untreated and alkali-treated makino bamboo fibers were used as fillers to fabricate cementitious composites in the present study. Accordingly, the functional groups, crystallinity, surface morphology of the bamboo fiber, and amount of calcium hydroxide (CH) in cementitious composites were determined using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA), and scanning electronic microscopy (SEM). Furthermore, the influences of bamboo fibers modified with various alkali treatments on the apparent density, drying shrinkage, and mechanical strength of cementitious composites were further investigated.

Materials and methods

Materials

Bamboo shavings from 3-year-old *Phyllostachys makinoi* (makino bamboo) were obtained by the local bamboo-processing factory (Nan-Tou county, Taiwan). These shavings were dried, hammer-milled and sieved to prepare bamboo fibers between 6 and 16 mesh. Type I ordinary Portland cement (Advanced-Tek Systems Co., Ltd., Taiwan) was used as a binder (2500–3300 kg/m^3). Local river sands as fine aggregates (a content of particles less than 75 µm of 2.2%) were purchased from Ligang Township in Taiwan. Sodium hydroxide (NaOH) were supplied from Merck KGaA (Darmstadt, Germany) to modify bamboo fibers.

Alkali treatment

Dried bamboo fibers were submerged in 5% and 10% NaOH solutions at 26 ± 2°C for 1, 12 and 24 h. After alkali treatment, the bamboo fibers were washed with distilled water to obtain alkali-treated bamboo fibers (AFs). Then, the AFs were stored at 20°C and 65% relative humidity (RH). The AFs with various alkali treatments are denoted as AF_{XY}, where X and Y are the NaOH concentration and treatment time (Table 1), respectively.

Preparation of cementitious composites

According to CNS 3655, cementitious composites with AFs (CAFs) were prepared and cured. In the mix design, the sand-cement and water-cement ratios were 3 and .5, respectively. AFs (5 wt%) with various alkali treatments were added to replace the mass fraction of cement in the present study. First, a uniform slurry was made by mixing cement and water at a stirring speed of 146 rpm for 15 sec. Second, sand and AFs were blended with the slurry to make fresh CAFs at a stirring speed of 313 rpm for 30 sec. The fresh CAFs were immediately cast for test specimens in different molds. Afterward, the test specimens were cured in saturated lime water for 28 days. The CAFs with various alkali-treated AFs are denoted as CAF_{xy}, where x and y are the NaOH concentration and treatment time (Table 1), respectively.

Table 1. The sample codes of the alkali-treated bamboo fibers (AFs) and the cementitious composites with AFs (CAFs).

	Sample code						
	AF_{NT} CAF_{NT}	AF_{0501} CAF_{0501}	AF_{0512} CAF_{0512}	AF_{0524} CAF_{0524}	AF_{1001} CAF_{1001}	AF_{1012} CAF_{1012}	AF_{1024} CAF_{1024}
NaOH concentration (%)	–	5	5	5	10	10	10
Treatment time (h)	–	1	12	24	1	12	24

NT:Untreated condition.

Testing procedures

Fourier transform infrared spectroscopy (FTIR)

FTIR spectra of AFs with various alkali treatments were determined to estimate their functional groups using a Perkin Elmer Spectrum 100 FTIR–ATR spectrophotometer. The spectra were recorded in the 650–4000 cm^{-1}, with a resolution of 4 cm^{-1} and accumulation of 32 scans.

X-ray diffraction (XRD)

XRD patterns of AFs with various alkali treatments were measured on a MAC science M×P18instrument (Japan). Additionally, the Ni-filtered $CuK_{\alpha 1}$ radiation (λ) is 0.1542 nm. The 2θ range was scanned at a scan rate of 2°/min in the ranges of 5–40°. The crystallinity index (CrI) of an AF was calculated as follows:

$$\mathrm{CrI}(\%) = (1 - I_{am}/I_{200})100 \tag{1}$$

where I_{200} is the intensity of the 200 lattice reflection at 2θ = 22.0° and I_{am} is the intensity of the amorphous material at 2θ = 18.4°.

Thermogravimetric analysis (TGA)

The amount of calcium hydroxide (CH) in CAFs with various AFs was estimated by a Pyris 1 TGA instrument (Perkin Elmer, Shelton, CT, USA). A total of 10 ± 1 mg of a specimen was heated at a constant heating rate of 10°C/min from 50 to 750°C in a nitrogen atmosphere (20 ml/min). The CH amount was calculated from the TG curves as follows:

$$\mathrm{CH}(\%) = \mathrm{WL_{CH}}(\%) \times (\mathrm{MW_{CH}}/\mathrm{MW_{H}}) \tag{2}$$

where WL_{CH} is the weight loss during CH hydration in the range of 400–500°C, MW_{CH} is the molecular weight of CH, and MW_H is the molecular weight of water.

Scanning electronic microscopy (SEM)

Morphologies of AFs with various alkali treatments were obtained from SEM micrographs using a Hitachi TM–1000 (Tokyo, Japan) with an acceleration voltage of 15 kV.

Mass loss (ML)

The bamboo fibers before and after alkali treatment were dried at 105°C for 2 days. Additionally, the *ML* values of dried AFs with various alkali treatments were calculated as follows:

$$ML(\%) = (1 - m_a/\mathrm{m}_0)100 \tag{3}$$

where m_0 and m_a are the masses of the fibers before and after alkali treatment, respectively.

Equilibrium moisture content (EMC)

All the AFs were placed at 25 ± 1°C and 65 ± 1% RH for 28 days. Afterward, the AFs were dried at 105°C for 2 days. The *EMC* values were determined as follows:

$$EMC(\%) = (m_c - m_o)/m_o 100 \tag{4}$$

where m_c and m_o are the masses of the AFs after conditioning and oven drying, respectively.

Apparent density

The volumes and mass of the 28-day-cured CAFs with dimensions of 50 mm × 50 mm × 50 mm were measured to calculate the apparent density (ρ) as follows:

$$\rho\left(\mathrm{kg/m^3}\right) = m/V \tag{5}$$

where m is the mass of the CAF (kg) and V is the volume of the CAF (m^3).

Drying shrinkage (DS)

The shrinkage tests for the CAF specimens with dimensions of 25 mm × 25 mm × 285 mm were carried out according to CNS 11,056. The shrinkages were measured using a digital length comparator (HCH–129, Jin Ching Her., Co., Ltd., Taiwan) at 23.0 ± 1.0°C in two humidity environments (75 ± 5% RH and 50 ± 1% RH) after 7, 14, 21, and 28 days. The DS values of the CAFs with various AFs were calculated as

$$DS(\%) = 100(L_x - L_o)/L_{ref} \tag{6}$$

where L_x is the length of the CAF specimen at drying time x (mm), L_o is the length of the CAF specimen before drying (mm), and L_{ref} is the length of the reference (= 250 mm).

Mechanical strength of CAFs

The tensile strength (TS) of briquet specimens for the CAFs was assessed with a span of 75 mm at a loading speed of 5 mm/min and according to ASTM C307. Additionally, the compressive strength (CS) was obtained using cube specimens (specimen size: 50 mm × 50 mm × 50 mm) at a tensile speed of 3 kgf/cm^2/sec according to CNS 1010. On the other hand, the modulus of rupture (MOR) of CAFs with dimensions of 40 mm × 40 mm × 160 mm was determined using a three-point static flexural test according to ASTM C348. During the flexural test, the flexural speed and support length were 1 kgf/cm^2/sec and 120 mm, respectively.

Analysis of variance

The properties of CAFs with different AFs were compared by Scheffe's test using SPSS (Statistical Product and Service Solutions) statistical software at a significance level of 5%.

Results and discussion

Characteristics of alkali-treated bamboo fibers (AFs)

Functional groups

Figure 1 presents the FTIR spectra of alkali-treated bamboo fibers (AFs) with different alkali treatments. To estimate the efficiency of alkali treatment, all spectra were normalized at 1030 cm^{-1}, which was assigned to C – O deformation in primary alcohols, aromatic C – H in-plane deformation, and

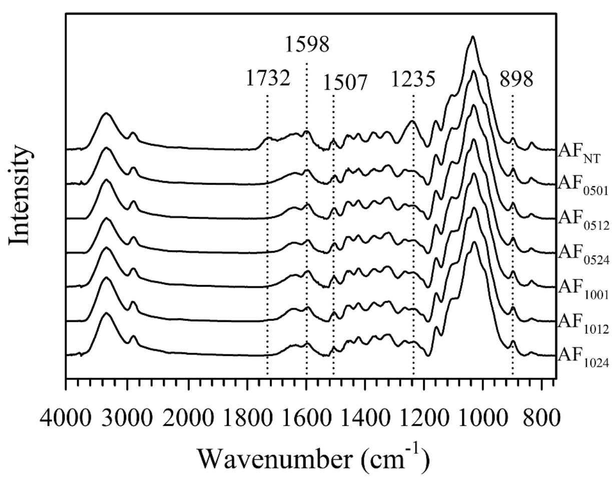

Figure 1. The FTIR spectra of the alkali-treated bamboo fibers (AFs).

C=O stretching (Chien et al. 2018). The band of the C=O stretching in hemicellulose appeared at 1732 cm^{-1} for untreated AFs (AF_{NT}). However, this band intensity of all AFs disappeared upon alkali treatment, which meant that the hemicellulose had been removed (Yang, Hua, and Hu 2023). The aromatic skeletal stretching in lignin for the AF_{NT} were at 1598 and 1507 cm^{-1}. Additionally, the band corresponding to C – O, C – C, and C=O stretching in lignin and xylan appeared at 1235 cm^{-1}. For the AFs, these band intensities decreased with increasing NaOH concentration and treatment time. Additionally, dramatic changes in the intensities of these peaks were observed for AF_{1024} (10% NaOH for 24 h). This result revealed that the lignin and hemicellulose were partially decomposed after alkali treatment. Furthermore, the asymmetric ring stretching band of cellulose at 898 cm^{-1} for the AFs slightly increased compared with the AF_{NT}. This phenomenon can be mainly explained by the fact that the removal of hemicellulose and lignin resulted in an increase in the relative percentage of cellulose during alkali treatment. Similar results were reported in the work of several researchers (Chattopadhyay et al. 2011; Das and Chakraborty 2006; Lin et al. 2018; Li, Zhou, and Guo 2017; Yang, Hua, and Hu 2023).

X-ray diffraction (XRD) patterns

The XRD patterns of the bamboo fibers treated with various NaOH concentrations and treatment times are shown in Figure 2. The crystallinity indices (CrIs) of the untreated and NaOH-treated bamboo fibers were calculated by the XRD pattern. The major cellulose crystal diffraction for the untreated bamboo fiber (AF_{NT}) appear at approximately 15.9° (110/1−10) and 22° (200), whereas a diffraction peak was found at approximately 34.7° (040). Additionally, the diffraction value of the amorphous cellulose region was at 18.4°. The diffraction patterns of the fibers treated with various NaOH treatments are similar to those of untreated fibers, and a significant shift of these characteristic peaks was not found. After lower NaOH concentrations (5% NaOH) at various treatment times, their CrI values were in the range of 52.9–54.7%, which were higher than that of AF_{NT} (49.4%). The increase in the CrI value is mainly attributed to hemicellulose and lignin reacting with alkali and being removed, which increases the proportion of cellulose with crystalline regions, thereby increasing the CrI value (Borchani, Carrot, and Jaziri 2015; Jayaramudu et al. 2011). When the NaOH concentration reached 10%, the CrI values of the AFs with various treatment times were in the range of 48.2–49.0%, which were similar to that of the AF_{NT} (49.4%). This finding may be explained by the interplay of two conditions. One is the increase in crystallinity due to the degradation and removal of cementing materials (hemicellulose, lignin, and amorphous regions in cellulose) (Borchani, Carrot, and Jaziri 2015; Jayaramudu et al. 2011). The other condition is related to partial transition of the cellulose structure. Liu and Hu (2008) reported that the cellulose structure may swell and relax with higher NaOH concentrations, and hydroxide ions further penetrate inside cellulose and undergo transition of

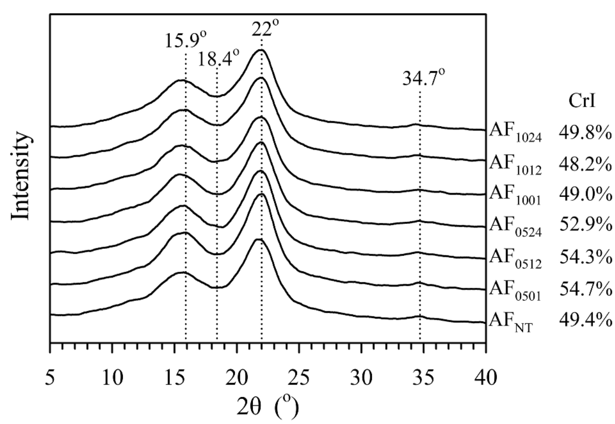

Figure 2. The XRD spectra and crystallinity (CrI) of the alkali-treated bamboo fibers (AFs).

the cellulose structure, resulting in the transition of cellulose I into II. Previous studies indicated that this cellulose transition resulted in a decrease in crystallinity (Chen et al. 2016; Ouajai and Shanks 2005). Taking these conditions into account, there were no significant changes in the crystallinity of the AFs with 10% NaOH at various treatment times, which could be a counterbalance of the removal of cementing materials and partial transition of cellulose structure.

Morphology of AFs

The microstructure of the alkali-treated bamboo fibers (AFs) with different alkali treatments using SEM images is shown in Figure 3. For the untreated bamboo fibers (AF_{NT}), it can be observed that the morphology of the untreated bamboo fiber (AF_{NT}) was straight with parenchyma cells on the surface of the AF_{NT} (Figure 3 a). After NaOH treatment, parenchyma cells were completely removed regardless of NaOH concentration and treatment time. At the given NaOH concentration, the fibers were more separated, disintegrated, and twisted with increasing treatment time, especially above 12 h (Figure 3 d–g). Additionally, the middle lamellae between fibers for 10% NaOH was more severe than that for 5% NaOH. Adel Salih et al. (2020) and Chen et al. (2021) stated that the fibers were significantly separated and twisted with increasing NaOH concentration since hemicellulose and lignin were more removed in the middle lamellae between fibers. This result indicated that the fibers were more obviously influenced with increasing treatment time, particularly when the NaOH concentration reached 10%.

Physical properties of AFs

The effects of treating the bamboo fibers with various concentrations of NaOH and treatment times on their mass loss (ML) and equilibrium moisture content (EMC) are shown in Table 2. The ML values of 5% NaOH solution-treated fibers are 10.15% for 1 h (AF_{0501}), 10.32% for 12 h (AF_{0512}), and 16.62% for 24 h (AF_{0524}), while the ML values of 10% NaOH solution-treated fibers are 10.72% for 1 h (AF_{1001}), 15.70% for 12 h (AF_{1012}), and 20.26% for 24 h (AF_{1024}). As expected, the ML values of the 10% NaOH-treated fibers are higher than those of the 5% NaOH-treated fibers, especially for longer treatment times (24 h). As the NaOH concentration and treatment time increased, the ML values of the AFs increased. According to the results of the FTIR spectra (Figure 1), the reduction in the mass of fibers is attributed to the degradation and removal of alkali-sensitive materials (hemicellulose and lignin) in bamboo fibers. Ray and Sarkar (2001) reported that there was a loss of 10.45% for jute fiber in a 5% NaOH solution within 2 h. At higher NaOH concentrations and longer treatment times (i.e., 10% at 24 h), the disintegration and degradation of cellulose caused a greater decrease in weight. Similar results were reported in the work of Das and Chakraborty (2007).

For the equilibrium moisture content (EMC) in Table 2, the EMC value of the AF_{NT} is 7.56%. After NaOH treatment, the EMC value of NaOH-treated fibers was higher than that of the AF_{NT}. At the given NaOH concentration, the EMC values of the 5% NaOH-treated fibers slightly increased from 8.50% (AF_{0501}) for 1 h to 8.62% for 24 h (AF_{0524}), whereas the EMC values of the 10% NaOH-treated fibers increased from 8.59% for 1 h (AF_{1001}) to 9.14% for 24 h (AF_{1024}). The results indicated that the EMC value increased with increasing NaOH concentration and treatment time. According to a previous study (Chen et al. 2018; Chowdhury et al. 2013; Young 1976), this finding could be related to the combined effects of the surface roughness of fibers, the accessibility of hydroxyl groups and the transition of the cellulose structure, further affecting the wettability and moisture content. From the results of the FTIR spectra (Figure 1), XRD spectra (Figure 2), and SEM images (Figure 3), a decrease in the accessibility of hydroxyl groups and an increase in the surface roughness were observed due to the removal of hemicellulose and lignin during alkali treatment. At lower NaOH concentrations (5% NaOH), the surface roughness may mainly affect the wettability of fibers, which might explain why the EMC values of treated fibers were higher than those of untreated fibers. Therefore, the surface roughness of the 5% NaOH-treated fibers increased with increasing treatment time, causing more wettability. When the NaOH concentration increased to 10%, the effect of the transition of the cellulose structure and surface roughness on the wettability of the fibers may overwhelm the

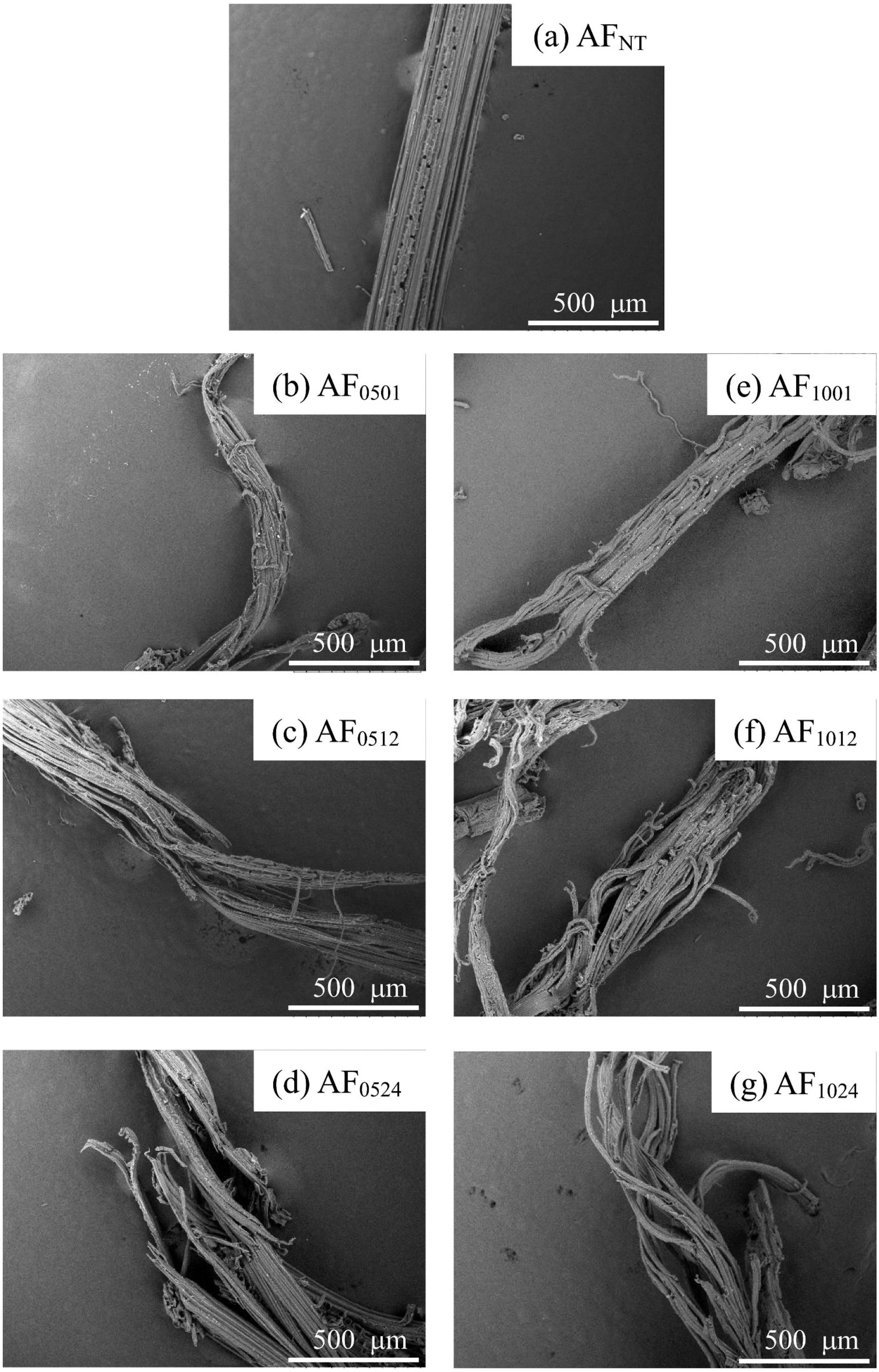

Figure 3. The SEM images of the alkali-treated bamboo fibers (AFs).

Table 2. The mass loss (ML) and equilibrium moisture content (EMC) of the alkali-treated bamboo fibers (AFs).

	AF_{NT}	AF_{0501}	AF_{0512}	AF_{0524}	AF_{1001}	AF_{1012}	AF_{1024}
ML (%)	–	10.15	10.32	16.62	10.72	15.70	20.26
EMC (%)	7.56	8.50	8.62	8.65	8.59	8.68	9.14

NT: Untreated condition.

accessibility of hydroxyl groups. As discussed in the results of XRD patterns, partial transition of cellulose I into II for the 10% NaOH-treated fibers was observed. Ishikawa et al. (1997) reported that cellulose II had a higher water absorption ability than cellulose I. The combination of these factors resulted in a further increase in the EMC value, and bamboo fibers with 10% NaOH at 24 h (AF_{1024}) had the highest EMC value among all specimens.

Characteristics of cementitious composites with AFs (CAFs)

Mineralogical change of cement in CAF

The effects of alkali-treated fibers on cement hydration were investigated by means of TGA results, which were obtained from cement materials that were hydrated for 28 days. As shown in Figure 4, three stages are found for all CAFs in each residual weight (RW) and differential RW curve. During the first stage of weight loss in the range of 50–200°C, water emission from the C-S-H gel and ettringite occurred (Chakraborty, Kundu, Roy, Adhikari, et al. 2013). The second and third stages correspond to the decomposition of calcium hydroxide ($Ca(OH)_2$, CH) and calcium carbonate phases at temperatures from 420–450°C and 600–700°C, respectively (Gabrovšek, Vuk, and Kaučič 2006). In Figure 4b, the CH amount of the untreated CAF (CAF_{NT}) is 2.25%. For the alkali-treated CAFs, their CH amounts of cement pastes are in the ranges of 2.69–2.90% and 2.79–3.33% for adding 5% and 10% NaOH-treated fibers, respectively. Jo and Chakraborty (2015) revealed that the higher weight loss in the second stage (the decomposition of CH) is representative of more CH produced by the cement hydration reaction. Therefore, the results in the present study showed that the CH amounts of all CAFs were higher than that of CAF_{NT}, which means that adding the AFs improved the hydration retardation effect of cement pastes. Additionally, the CH amounts of the CAFs increased with increasing NaOH concentration and treatment time. Similar investigations were reported in the work of Jo and Chakraborty (2015). They reported that the cement hydration reaction was retarded due to adding untreated natural fibers into cement pastes, while natural fibers treated by NaOH reduced the hydration retardation effect (Jo and Chakraborty 2015). Natural fibers are in a highly alkaline environment during the hydration reaction of cement, causing the degradation and leaching of organic components in natural fibers, such as hemicellulose and lignin. These organic components either chelate with the cations present in the hydrated cement or form a barrier layer around the partial cement grain to delay the cement hydration reaction and hinder the nucleation and growth of the hydrated cement product (Bishop and Barron 2006; Chakraborty, Kundu, Roy, Adhikari, et al. 2013; Jo and Chakraborty 2015). In the present study, more organic components were removed from the fibers as the NaOH concentration and treatment time increased, further reducing the hydration retardation effect.

Drying shrinkage of CAFs

Figure 5 shows the drying shrinkage (DS) development of cementitious composites with alkali-treated bamboo fibers (CAFs) under different relative humidities (RHs) at various drying periods. Additionally, Tables 3 and 4 present the significant differences among the DS values of the CAFs under different drying processes in the statistical analysis. Under 75% RH (Figure 5a and Table 3), the DS value of CAF_{NT} rapidly increases within the first 21 days since large water evaporation occurs in the early period, after which its shrinkage rate slows down. Moreover, the DS values of CAFs notably decrease by adding alkali-treated fibers. The higher DS value for the CAF_{NT} can be explained by the increased water introduced by adding hydrophilic bamboo fibers into the cement, which results in

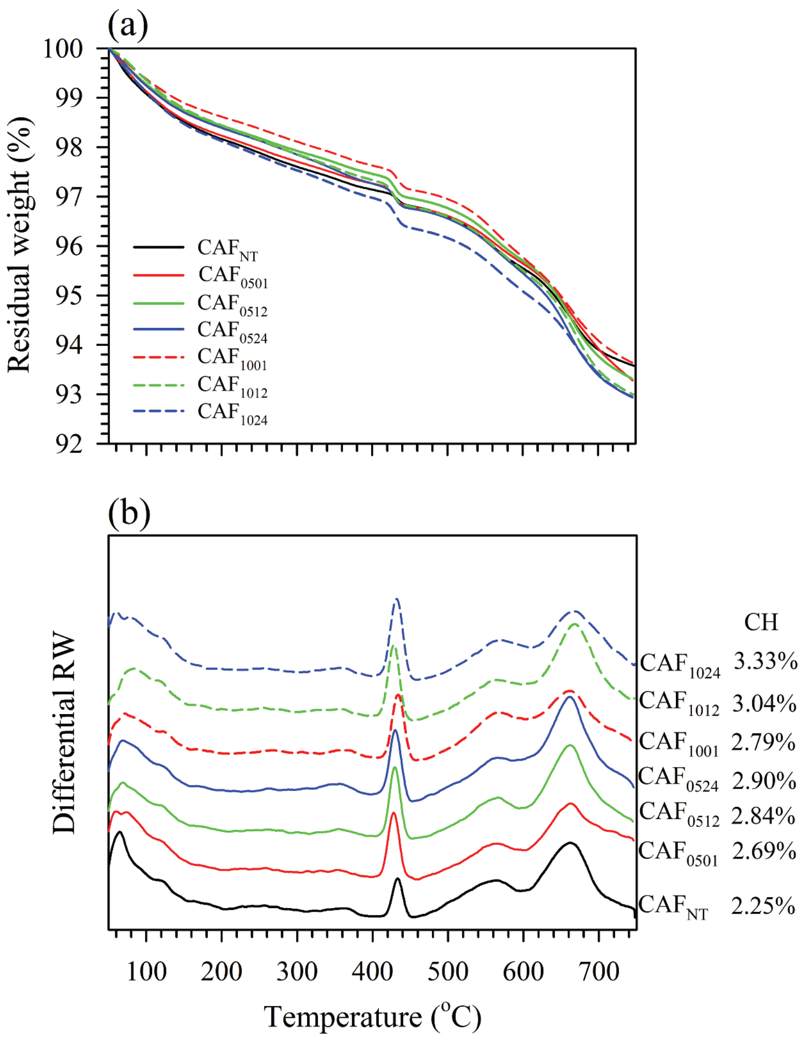

Figure 4. The thermogravimetric curves and the amount of calcium hydroxide (CH) of the cementitious composites with alkali-treated bamboo fibers (CAFs). (a) The curves of residual weight (RW); (b) the curves of differential RW.

a higher evaporated water loss, thus causing larger drying shrinkage. The other explanation is that the porosity of the bamboo itself, the porosity induced by the bamboo in the cement and the connectivity of pores (Bederina et al. 2012; Mohr, Biernacki, and Kurtis 2006; Roma, Martello, and Savastano 2008), accelerate the drying process and consequently increase desiccative deformation (X. Zhang et al. 2021). Additionally, under higher RH (75% RH), the DS values of the CAFs with alkali-treated fibers decreased by 21.9–34.2% at a longer drying period (28 days) compared to those with untreated fibers (Table 3). One reason can be that alkali treatment decreased the hydrophilicity of the bamboo fibers to improve the dimensional stability, which is influenced by water. Another reason may be that the alkali-treated fibers have a higher surface area and a greater number of fibers to bond with the cement to enhance the bonding between the fiber and the cement matrix, thus reducing drying shrinkage (Guo, Sun, and Satyavolu 2020).

The other important parameter is the external RH, which affects moisture transport from cement to the environment (Bissonnette, Pierre, and Pigeon 1999). As presented in Figure 5b and Table 4, the DS values of CAFs with alkali-treated bamboo fibers under 50% RH at various drying periods were

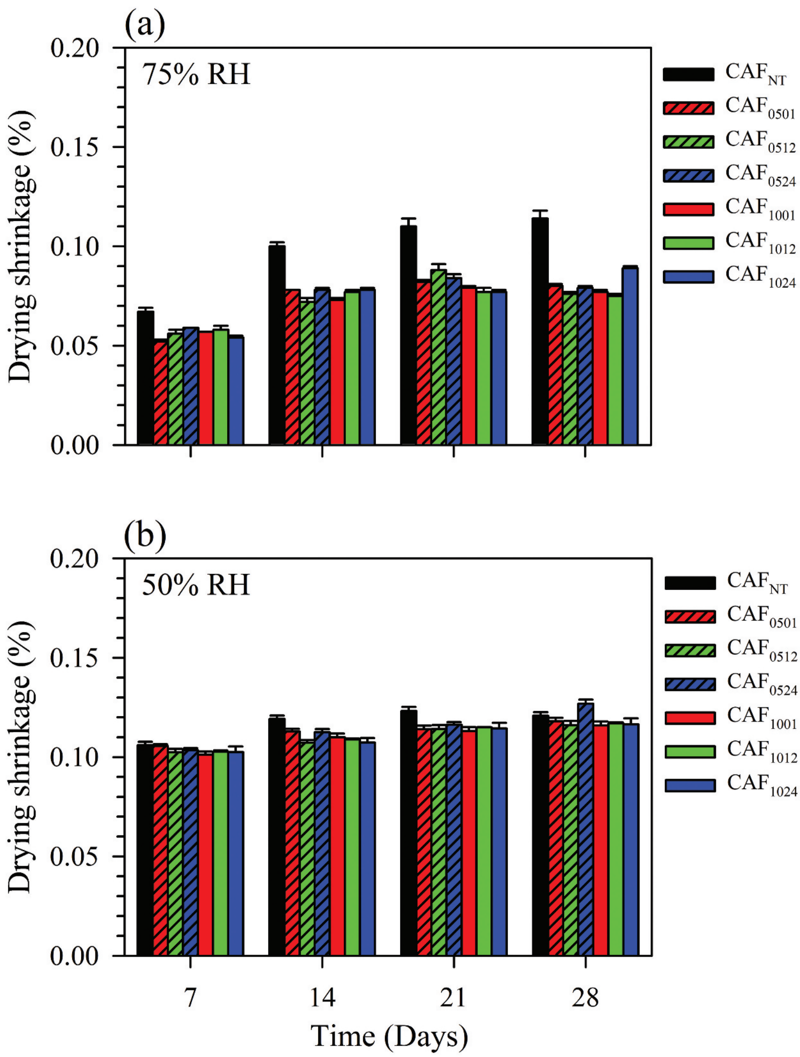

Figure 5. The drying shrinkage of the cementitious composites with alkali-treated bamboo fibers (CAFs) under different humidities at various drying periods. (a) 75% RH; (b) 50% RH.

Table 3. The drying shrinkage (DS) of cementitious composites with alkali-treated bamboo fibers (CAFs) under 75% RH at different drying periods.

	DS (%)			
Code	7 days	14 days	21 days	28 days
CAF_{NT}	$0.067 \pm 0.002^{a,C}$	$0.100 \pm 0.002^{a,B}$	$0.110 \pm 0.004^{a,A}$	$0.114 \pm 0.004^{a,A}$
CAF_{0501}	$0.052 \pm 0.001^{d,C}$	$0.078 \pm .000^{b,B}$	$0.082 \pm 0.001^{bc,A}$	$0.080 \pm 0.001^{c,A}$
CAF_{0512}	$0.056 \pm 0.002^{bcd,C}$	$0.072 \pm 0.002^{d,B}$	$0.088 \pm 0.003^{b,A}$	$0.076 \pm 0.001^{c,B}$
CAF_{0524}	$0.059 \pm 0.000^{b,C}$	$0.078 \pm 0.001^{b,B}$	$0.084 \pm 0.002^{bc,A}$	$0.079 \pm 0.001^{c,B}$
CAF_{1001}	$0.057 \pm 0.000^{bc,C}$	$0.073 \pm 0.001^{cd,B}$	$0.079 \pm .001^{c,A}$	$0.077 \pm 0.001^{c,A}$
CAF_{1012}	$0.058 \pm 0.002^{bc,B}$	$0.077 \pm 0.001^{bc,A}$	$0.077 \pm 0.002^{c,A}$	$0.075 \pm 0.001^{c,A}$
CAF_{1024}	$0.054 \pm 0.001^{cd,C}$	$0.078 \pm 0.001^{b,B}$	$0.077 \pm 0.001^{c,B}$	$0.089 \pm 0.001^{b,A}$

Values are the mean ± SD ($n = 3$). Lowercase letters indicate significant differences among different groups at a given drying period. Capital letters indicate significant differences among different drying periods in a given group.

Table 4. The drying shrinkage (DS) of cementitious composites with alkali-treated bamboo fibers (CAFs) under 50% RH at different drying periods.

Code	DS (%)			
	7 days	14 days	21 days	28 days
CAF_{NT}	$0.106 \pm 0.002^{a,B}$	$0.119 \pm 0.002^{a,A}$	$0.123 \pm 0.002^{a,A}$	$0.121 \pm 0.002^{ab,A}$
CAF_{0501}	$0.106 \pm 0.001^{a,C}$	$0.113 \pm 0.001^{ab,B}$	$0.114 \pm 0.002^{b,AB}$	$0.118 \pm 0.002^{b,A}$
CAF_{0512}	$0.102 \pm 0.002^{a,B}$	$0.107 \pm 0.001^{b,B}$	$0.114 \pm 0.002^{b,A}$	$0.116 \pm 0.002^{b,A}$
CAF_{0524}	$0.104 \pm 0.001^{a,C}$	$0.113 \pm 0.002^{b,B}$	$0.116 \pm 0.001^{b,B}$	$0.127 \pm .002^{a,A}$
CAF_{1001}	$0.101 \pm 0.002^{a,C}$	$0.110 \pm 0.002^{b,B}$	$0.113 \pm 0.002^{b,AB}$	$0.116 \pm 0.002^{b,A}$
CAF_{1012}	$0.103 \pm 0.001^{a,C}$	$0.109 \pm 0.004^{b,B}$	$0.115 \pm 0.000^{b,A}$	$0.117 \pm 0.001^{b,A}$
CAF_{1024}	$0.103 \pm 0.003^{a,C}$	$0.107 \pm 0.002^{b,BC}$	$0.114 \pm 0.003^{b,AB}$	$0.117 \pm 0.003^{b,A}$

Values are the mean ± SD ($n = 3$). Lowercase letters indicate significant differences among different groups at a given drying period. Capital letters indicate significant differences among different drying periods in a given group.

investigated. Regardless of the CAFs with various NaOH concentrations and treatment times, the DS values under 50% RH at various drying periods were higher than those under 75% RH. Additionally, there were much fewer differences in the DS values under 50% RH between the CAFs with alkali-treated fibers and the CAF_{NT} compared to 75% RH. Furthermore, the DS values under 50% RH for all specimens more rapidly increase within the first 14 days. Previous studies have reported that changes in RH form drying-induced stresses in cement, producing different degrees of shrinkage and damage (Gao et al. 2020; Samouh, Rozière, and Loukili 2019). Especially at low RH, several mechanisms, such as capillary pressures, disjoining pressures, and pressures induced by interfaces, may be involved in drying shrinkage of the cement (Bentz, Garboczi, and Quenard 1998; Kovler and Zhutovsky 2006; Mindess, Young, and Darwin 2002; Yates 1954). Samouh et al. (2019) found that a larger DS value of cement occurred under approximately 50% RH since the maximum capillary and disjoining pressures were reached.

Density and mechanical strength of CAFs

As presented in Table 5, the density of the cementitious composites with untreated fibers (CAF_{NT}) was 2054 kg/m^3. At the lower NaOH concentration (5% NaOH), the density of the CAFs significantly decreased from 2032 kg/m^3 (CAF_{0501}) to 1994 kg/m^3 (CAF_{0524}) with increasing treatment time. Similarly, when the NaOH concentration reached 10%, the density of the CAFs was 2066 kg/m^3 for 1 h, 1986 kg/m^3 for 12 h, and 1950 kg/m^3 for 24 h. According to the results described above, there was no significant difference among the density of CAF_{NT}, CAF_{0501}, and CAF_{1001} in the statistical analysis. This occurrence of the above phenomenon is related to more hydration and carbonation of the cement pastes in CAF_{0501} and CAF_{1001} compared to CAF_{NT}. Moreover, the results show that the average density of the 5% NaOH- and 10% NaOH-treated CAFs at a treatment time of 24 h decreased by 2.9% and 5.1% of the CAF_{NT}. When the NaOH concentration was less than 10%, adding fibers treated for more than 1 h significantly impacted the density of CAFs. Das and Chakraborty (2007) observed that

Table 5. The density and mechanical strength of cementitious composites with alkali-treated bamboo fibers (CAFs).

Code	Density (kg/m^3)	TS (MPa)	CS (MPa)	MOR (MPa)
CAF_{NT}	2054 ± 18^{ab}	3.9 ± 0.3^{a}	28.7 ± 1.7^{a}	5.8 ± 0.2^{ab}
CAF_{0501}	2032 ± 14^{b}	3.4 ± 0.2^{a}	19.2 ± 0.6^{bc}	5.4 ± 0.3^{abc}
CAF_{0512}	1983 ± 25^{c}	3.6 ± 0.3^{a}	16.4 ± 0.8^{cd}	4.8 ± 0.4^{c}
CAF_{0524}	1994 ± 26^{c}	3.5 ± 0.4^{a}	2.9 ± 1.2^{b}	5.0 ± 0.3^{bc}
CAF_{1001}	2066 ± 14^{a}	3.7 ± 0.2^{a}	2.8 ± 1.0^{b}	5.8 ± 0.4^{a}
CAF_{1012}	1986 ± 26^{c}	3.5 ± 0.2^{a}	16.1 ± 1.3^{cd}	4.6 ± 0.2^{c}
CAF_{1024}	1950 ± 27^{d}	3.5 ± 0.2^{a}	15.8 ± 2.3^{d}	4.7 ± 0.8^{c}

Values are the mean ± SD ($n = 12$ and 6 for density and mechanical specimens, respectively). Lowercase letters indicate significant differences among different composites.

alkali treatment caused a gradual reduction in the density of bamboo fibers as the alkali concentration increased. The decrease in the density of bamboo fibers may be attributed to the removal of alkali-sensitive components (ex. hemicellulose and lignin). Therefore, compared to CAF_{NT}, CAFs with a lower density are related to adding bamboo fibers with a reduced density. Furthermore, due to the low density of alkali-treated fibers, the weight of treated fibers per unit volume was required to be higher to fabricate the CAF, especially for higher NaOH concentrations and treatment times (CAF_{1024}).

Table 5 gives the tensile strength (TS), compressive strength (CS), and modulus of rupture (MOR) of all the CAFs with untreated and alkali-treated BFs. The TS value of CAF_{NT} is 3.9 MPa, while the TS values of the alkali-treated CAFs are in the range of 3.4 to 3.7 MPa. In the statistical analysis, there were no significant differences in the TS values of the CAFs among all specimens, which implied that the tensile strength of the CAFs was not affected by material density. Generally, the tensile properties of natural fiber-added composites are mainly dependent on the tensile strength of the fibers and the interfacial adhesion between the fibers and matrix. No significant influence on the tensile strength of the CAFs in the present study might be attributed to the interplay of two factors. One factor is the reduction in tensile strength of fibers treated by NaOH solution. This phenomenon should be caused by the depolymerization of hemicellulose, lignin and amorphous cellulose and destruction of the lignin – hemicellulose copolymer in the natural fibers after a higher NaOH concentration or higher treatment period (Adel Salih, Zulkifli, and Azhari 2020; Wang et al. 2018; Zukowski, de Andrade Silva, and Toledo Filho 2018). The other factor is the higher surface roughness of NaOH-treated natural fibers, which achieves good adhesive interaction between the fibers and cement matrix. Zukowski et al. (2018) reported that the pull-out strength of cementitious composites with alkali-treated fibers was higher than that of composites with untreated fibers. This behavior is related to the greater surface roughness of the natural fiber after alkali treatment, which causes a higher frictional bond with the cement matrix. Additionally, the cement hydration products were filled in voids of the fiber surface that were created by alkali treatment to enhance mechanical interlocking and entanglement between fibers and cement, providing the frictional force and the effective transfer of the stress between them (Banjo Akinyemi, Temidayo Omoniyi, and Onuzulike 2020; Yan et al. 2016; X. Zhang et al. 2021). As described above, a significant offset from these factors might lead to no significant changes in the tensile strength of the CAFs with alkali-treated bamboo fibers.

However, a significant difference in the compressive strength (CS) and modulus of rupture (MOR) was observed with the TS results. As shown in Table 5, the CS values of all alkali-treated CAFs significantly decreased by 27.2–44.9% compared with the CAF_{NT}. At lower NaOH concentrations (5% NaOH), the CS value decreased from 28.7 MPa (CAF_{NT}) to 16.4 MPa with increasing treatment time up to 12 h. When the treatment time was increased to 24 h (CAF_{0524}), the CS value showed a slight increase to 20.9 MPa. Additionally, the CS values of the 10% NaOH-treated CAFs significantly decreased from 20.8 MPa (CAF_{1001}) to 15.8 MPa (CAF_{1024}) with an increase in treatment time. Comparing different NaOH concentrations, there are significant differences among the CS values of the CAFs with fibers treated at 24 h. These behaviors could be related to the combination of the density of the CAFs and fiber strength. A decrease in the CS value is caused by density reduction of the CAFs due to the addition of alkali-treated fibers with low mass. Furthermore, the lower CS value shown by NaOH-treated specimens could possibly be caused by the weakened fibers. Similarly, the MOR results of CAFs are consistent with the CS values (Table 5). At a lower treatment time (1 h), the MOR values of the CAFS were not significantly different from those of CAF_{NT} (5.8 MPa). However, the MOR values distinctly decreased as the treatment time increased from 1 to 24 h, especially for higher NaOH concentrations (10% NaOH). Regardless of NaOH concentration, the MOR values of the alkali-treated CAFs significantly decreased above 1 h of treatment time compared with the CAF_{NT}. This may be attributed to a reduction in the density of the CAFs and lower strength of the fibers, which could decrease the MOR values of the alkali-treated CAFs. As described above, the TS values of all alkali-treated CAFs exhibited no significant differences when compared with those of CAF_{NT}, while the MOR and CS values were seriously affected by NaOH treatment.

Conclusions

The influences of adding alkali-treated bamboo fibers (AFs) on the properties of cementitious composites with AFs (CAFs) were investigated in the present study. For functional groups of the AFs using FTIR, the lignin and hemicellulose were partially decomposed after alkali treatment, especially for the higher concentration and treatment time (i.e., 10% NaOH and 24 h of treatment). For crystallinity indices (CrIs) calculated by the XRD pattern, the CrI values of the 5% NaOH-treated AFs at various treatment times (52.9–54.7%) were higher than those of untreated bamboo fibers (49.4%). When the NaOH concentration reached 10%, the CrI values of the AFs with various treatment times (48.2–49.0%) were similar to those of untreated bamboo fibers. Additionally, the SEM images for the morphology of the AFs were more separated, disintegrated and twisted with increasing NaOH concentration and treatment time, while the mass loss and equilibrium moisture content increased. Furthermore, the TGA results showed that the amounts of calcium hydroxide (CH) in all CAFs were higher than those in untreated CAFs, and they increased with increasing NaOH concentration and treatment time. For drying shrinkage (DS) under 75% RH, the DS values of the CAFs notably decrease by adding AFs. However, there were fewer differences in the DS values under 50% RH compared to the untreated CAFs. The average density of the 5% NaOH- and 10% NaOH-treated CAFs with longer treatment times (i.e., 24 h) decreased by 2.9% and 5.1% compared to the density of the untreated CAFs, respectively. For the mechanical strength of the CAFs, there were no significant differences in the tensile strength of the CAFs among all specimens, while significant reductions in the compressive strength and modulus of rupture for all alkali-treated CAFs were observed compared with those of the untreated CAFs. As described above, these results indicated that the alkali-treated bamboo fibers significantly improved the drying shrinkage of the CAFs and hydration retardation effect of cement pastes, while the density, flexural strength and compressive strength of the alkali-treated CAFs decreased.

Highlights

- Effects of adding alkali-treated bamboo fibers (AFs) on the properties of cementitious composites with AFs (CAFs) were investigated.
- Bamboo fibers were treated with various NaOH concentrations and treatment times.
- AFs significantly improved the drying shrinkage of the CAFs and hydration retardation effect of cement pastes.
- A decrease in density, flexural strength and compressive strength of the alkali-treated CAFs was observed.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This work was supported by the National Science and Technology Council, Taiwan under Grant NSTC 108-2313-B-005-014-MY3 and NSTC 111-2313-B-005-028-MY3.

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Author's contribution

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