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Water absorption, tensile, flexural and impact properties of aged bamboo fibre/nano $CaCO_3$-modified unsaturated polyester composites

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This paper presents the investigation on the effects of nano $CaCO_3$ on the durability of bamboo fibres/unsaturated polyester composites aged in distilled water up to 112 days. The nano $CaCO_3$ contents in the unsaturated polyester matrix were 0, 1, 3 and 5 wt%, while the bamboo fibre content in the bamboo fibre/nano $CaCO_3$-modified unsaturated polyester composites was fixed by 25 vol%. It was found that the equilibrium water uptake and diffusion rate decreased up to 16.2 and 15.6 %, respectively, by adding nano $CaCO_3$ up to 3 wt%; however, at the content of 5 wt%, they were higher than those of the unfilled composites. For the unaged composites, the tensile and flexural strengths were improved by adding nano $CaCO_3$ up to 1 and 3 wt%, respectively, whereas adding 5 wt% could not give positive effects. On the other hand, the increase of nano $CaCO_3$ up to 5 wt% increased the elastic modulus almost linearly for both loadings. For the aged composites, adding 1-5 wt% nano $CaCO_3$ reduced the degradation of composites both in tensile and flexural loadings, but the best enhancement was obtained at the content range of 1-3 wt%. Under impact loading, adding 1-5 wt% nano $CaCO_3$ enhanced the impact strength of composites, regardless of the ageing condition.

Keywords: bamboo fibre, unsaturated polyester, nano $CaCO_3$, durability, composites

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1. Introduction

Natural fibres have been increasingly used for manufacturing green composite materials providing lightweight, and environmentally friendliness. However, natural fibres absorb much water and affect the integrity of structures made of the natural fibre composites. It has been reported that chemically fibre treatments could reduce the water absorption of composites by improving the fibre-matrix bonding. Such treatments included alkali treatment [1, 2], silane treatment [3], isocyanatoethyl methacrylate [4], benzoyl and benzyl chloride [5].

Besides treating the fibres, modifying the matrix can also reduce the absorbed water in the natural fibre-reinforced polymeric composites. Inorganic nanofillers are most utilised to reduce the water absorption of the natural fibre-reinforced polymeric matrix. Nanofillers increased the contact surface area and increased the tortuosity of water diffusion into the polymer [6]. Nano clay has been extensively used to modify epoxy matrix for manufacturing natural fibre nanocomposites [7]. For example, adding 5 wt% nano clay was able to decrease the water absorption of sisal/epoxy nanocomposites up to 80 % and improved the aged tensile properties [7]. A decrease of water absorption up to 60 % at the nano clay content of 5 wt% was also reported on the recycled cellulose fibre/vinyl-ester composites [8]. Besides nano clay, hard particles such as nano silicon carbide (SiC) are also potential to reduce water absorption. At the content of 5 wt%, nano SiC decreased the water absorption in the recycled cellulose/epoxy composites up to 47 % and reduced the mechanical properties degradation of the composites due to ageing [9].

Relatively low-cost nanofiller such as nano CaCO3

(NCC) has also been increasingly used to modify polymer for manufacturing fibrous composites. A small amount of NCC up to 4 wt% could increase the thermal properties of nanocomposites by increasing glass transition temperature from 81 to 183 °C for epoxy [10], and from 93 to 120 °C for unsaturated polyester [11]. Adding NCC up to 4 wt% also increased the toughness and flexural properties of epoxy by about 44 % and 22 %, respectively [12]. The improved thermal and mechanical properties of polymer were caused by hindering the chains mobility in the polymer structures. The improvement of thermal and mechanical properties of NCC-modified epoxy also increased the thermal properties, and mechanical properties of the fibrous composites such as glass fibre- and carbon fibre-reinforced composites. For example, the addition of 8 wt% NCC increased the char residue of glass fibre/epoxy composites considerably, from 7.1 % of neat epoxy to 50.4 % of the NCC/glass fibre/epoxy composites [13]. On the mechanical properties, a continuous increase of flexural modulus and strength, fracture toughness and impact strength with the increase of NCC up to 8 wt% were reported by Zulfli et al. [13]. While He et al. [14] reported the improvement of compressive, strength, flexural strength and impact strength by about 13.5, 22.7 and 39.3 %, respectively, by adding 4 wt% NCC in the carbon fibre/epoxy composites. Another researcher, Eskizeybek et al. [15] studied the dynamic properties of carbon fibre/NCC-modified epoxy composites and reported that adding 2 wt% NCC improved the low-speed impact property by 16.8 % at the impact speed of 2 mm/s.

Bamboo fibre has gained much attention for manufacturing composites as bamboo is a fast-growing plantation, highly available and has high mechanical properties [16]. Several studies of NCC utilisation on the bamboo fibre reinforced polymer composites have been reported. Wang et al. [17, 18] used NCC to increase the tensile strength of individual bamboo fibre by impregnation method and using the method to compare with the blending method in manufacturing bamboo fibre/high-density polyethylene (HDPE) composites. The impregnation method provided a high degree of fibre-matrix interaction than the blending method in bamboo fibre/HDPE composites. For outdoor application, increasing durability of bamboo fibre composites is becoming the focus of research. Unsaturated polyester resin is still preferred to manufacture the outdoor bamboo fibre composites because it is low-cost resin and has low water absorption. However, unsaturated polyester resin often experienced leaching during long term ageing that further degraded their composites [2]; therefore, the addition of low-cost NCC might simultaneously reduce water absorption and improve the stability and durability of the composites. To the best authors knowledge, the durability of bamboo fibre/NCC-modified unsaturated composites is rarely reported. This paper is aimed to investigate the effects of NCC content on the durability of bamboo fibre/unsaturated polyester composites. Water absorption was studied using gravimetric method along with the Fourier Transform Infrared spectroscopy (FTIR) to investigate the water-composites interaction. Scanning electron microscopy (SEM) was carried out to examine the fracture surfaces of broken specimens before and after being aged up to the saturation level.

2. Materials and method

2.1. Materials

The bamboo fibre was extracted from Gygantochloa Apus species. The bamboo culms were crumbed and combed to obtain bamboo fibre bundles, with the average diameter and length of 0.5 mm and 10 mm, respectively. The bamboo fibres were treated using sodium hydroxide solution (8 wt%) in a bath for 2 hours [19]. A general-purpose unsaturated polyester resin (UPR) was used, supplied by Eternal Co. Ltd (Taiwan) and methyl ethyl ketone was used as a hardener. Cubic-shaped NCC particles were purchased from BWChem (China) with the average particles size of 60-80 nm and a specific surface area of ≥ 20 m^2/g. The received NCC had been treated using fatty acid. The fatty acid treatment improved the compatibility with the polymeric matrix such as epoxy [20].

2.2. Specimen fabrication

Specimen fabrication was conducted using a compression moulding technique. The bamboo fibres were laid up randomly in the 15 cm × 30 cm sized steel mould. The volume fraction of bamboo fibre in the bamboo fibre/nano $CaCO_3$-modified UPR composites was fixed by 25 vol% [21], regardless of the nano $CaCO_3$ content in the UPR matrix. The unsaturated polyester resin was mixed with NCC, with the NCC contents of 0, 1, 3, and 5 wt%, before being used to manufacture the bamboo fibre/NCC-modified UPR composites. So, the NCC content was calculated based on the matrix only without bamboo fibre reinforcement. As most of the nanoparticles were able to well-dispersed in a polymeric matrix up the content of 5 wt%, so the NCC contents within that range were selected. Firstly, NCC was added in the unsaturated polyester resin and then stirred using a high-speed mixer at the speed of 10,000 rpm for 10 min. After that, the mixture was degassed in a vacuum chamber until the air bubbles were released from the mixture. The hardener was added and stirred slowly to avoid trapping the air bubbles. The weight ratio of resin to hardener was 100 : 0.25. The modified unsaturated polyester resin then was poured into the bamboo fibre-filled mould (at the middle), and then followed by compressing the mould using the male mould. Then the composites were cured at room temperature for 24 hours. The cured composites were cut to obtain the tensile, flexure, impact and water absorption specimens. The bamboo fibre/NCC-modified UPR composites with the NCC contents of 0, 1, 3 and 5 wt% were then designed as BF/UPR_NCC0, BF/UPR_NCC1, BF/UPR_NCC3, and BF/UPR_NCC5, respectively. The tensile, flexural and impact specimens of composites followed the ASTM D3039 [22], ASTM D790 [23], and ASTM D256 [24], respectively. The size of water uptake specimen was 52 mm × 52 mm × 4.5 mm. The tensile, flexural and impact specimens of pure UPR was also cast according to ASTM D638 [25], ASTM D790 [23], and ASTM D256 [24], respectively.

2.3. Water uptake study

The specimens for water uptake study (with three replications) were immersed in distilled water at room temperature. The samples weighing was conducted at every 3 hours on the first two days, and once on the following days, using a Kenko digital microbalance. The weighing was carried out until the saturation had been achieved. The water uptake (Mt) was calculated using Eq. (1)

$$M_t = \frac{W_t - W_o}{W_o} \times 100\% \tag{1}$$

where, W_t is the specimen weight after the ageing time t, and W_o is the initial weight. Curve fitting of Fickian diffusion law was conducted on the water uptake data, following the Eq. (2) [26].

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{2n+1}\exp\left[\frac{-(2n+1)^2\pi^2 Dt}{4l^2}\right] \tag{2}$$

The diffusion rate (D) was calculated based on the linear part of water uptake vs. square root of ageing time up to $M_t/M_\infty = 0.6$, using Eq. (3) [26].

$$D = \frac{\pi}{16}\left(\frac{m \times h}{M_\infty}\right)^2 \tag{3}$$

where m is the slope of the linear part of water uptake vs. square root of ageing time, h is the specimen thickness, and M_∞ is the equilibrium water uptake.

2.4. Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectroscopy was carried out using a PerkinElmer Frontier machine. The selected specimens were ground to obtain powder about 20 mg, mixed with KBr and then compacted. The unaged and aged (saturation level) samples were tested. A single measurement of 20 mg tested for each variation might not represent the whole samples; however, this FTIR test was intended as an indicative tool for the interaction between NCC and UPR matrix and the existence of water molecules (functional groups), which might not be critical as that for quantitative measurements.

2.5. Mechanical testing

The tensile and flexural tests were conducted using a 10 kN Tensilon universal testing machine, with the displacement speeds of 5 and 2 mm/min, respectively. The impact test was performed by using an Izod test method. At least four specimens were tested for the tensile, flexural and the impact testing. The specimens were grouped in unaged (dry) and aged (wet) conditions. For the aged specimens, the specimens were immersed in distilled water at room temperature. Testing for the aged specimens was conducted at two withdrawals. First and the second withdrawals were carried out at the ageing times of 24 and 112 days, respectively. The water content in the specimens of the first and second withdrawals simulated the start and long after the saturation level, respectively.

2.6. Morphology study

The morphologies of fracture surfaces were studied using an optical and a scanning electron microscopy (SEM). The SEM was carried out using a Phenom Pharos Desktop SEM machine, with the operating voltage of 5 kV.

3. Results and discussion

3.1. Water absorption

Fig. 1 shows the water uptake vs. square root of ageing time curves of the bamboo fibre/NCC-modified UPR composites, while the diffusion rate (D) and the equilibrium water uptake (M_∞) are shown in Table 1. Fickian fits were shown to check the diffusion behaviour. It was observed that the water uptake increased linearly with the square root of ageing time at the initial stage, and then slowed down approaching the saturation level. The water diffusion behaviour did not follow the Fickian diffusion model as the deviation occurred at the knee regions or it behaved as pseudo-Fickian [2]. It was seen that compared to the pure UPR, the equilibrium water uptake and diffusion rate of bamboo fibre/NCC-modified UPR composites increased considerably, in factor 6.1 – 7.6 and 4.4 – 5.2, respectively. The increase of water uptake properties of composites, compared to pure UPR was mostly attributed to bamboo fibres. Meanwhile, among the composites, the water uptake was mostly determined by the addition of NCC as the bamboo fibre content was constant. The water uptake and diffusion rate tended to decrease with the increase of NCC content up to 3 wt% and then increased again at the NCC content of 5 wt%. At the NCC content of 5 wt%, the water uptake and diffusion rate became higher than that at the content of 0 wt%. Compared to the NCC content of 0 wt%, the difference of water uptake at the NCC contents of 1, 3, and 5 wt% was about -11.4, -16.2, and +7.4 %, respectively, while the difference of diffusion rate was -6.5, -15.6 and +0,9%, respectively.

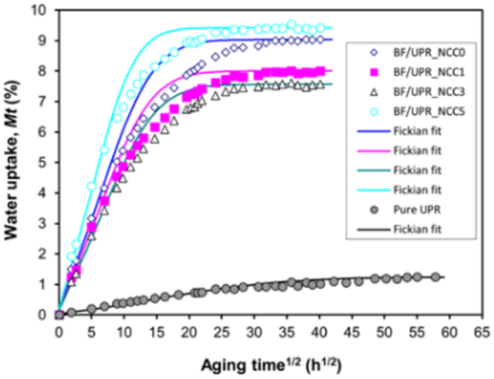

Fig. 1. The water uptake of bamboo fibre/NCC modified UPR composites.

Adding nanoparticles into thermoset polymers decreased the water absorption as well as the diffusion rate, as reported by other researchers [6, 27, 28]. The nanoparticles increased the tortuosity of water diffusion. Hence, the diffusion rate and the equilibrium water uptake decreased with the increase of NCC content up to a certain extent. In this study, the decrease of diffusion rate and the equilibrium water occurred up to the content of 3 wt%, which could be caused by the good dispersion of NCC. In contrast, for the content beyond 3 wt%, (i.e. at 5 wt%), the NCC might form clusters that increased the diffusion rate and the equilibrium water uptake again, as also reported by He et al. [27]. The clusters might then become the site for water to accumulate, increasing water absorption and the diffusion rate. The particles clustering will be clarified later in the SEM micrographs of fracture surfaces.

3.2. Fourier transform infrared spectroscopy

Fig. 2 shows the FTIR spectra of the unaged and aged bamboo fibre/NCC-modified UPR composites. The FTIR spectra of UPR, NCC and bamboo fibre are shown for comparison. For the unaged samples (Fig. 2a), the FTIR spectra of bamboo fibre/NCC-modified UPR composites did not differ significantly to that of pure UPR. The main absorbance peaks of pure UPR were at the wavenumbers of 3447, 1732, 1454, 1283, 1067 and 700 cm^{-1}. Those wavenumbers are associated to OH stretching of water or glycol, -C=O stretching of ester, C-H bending of alkane, C-O stretching of aromatic ester, C-O-C ester linkage, and C-H bending of benzene aromatic ring, respectively [29]. Adding bamboo fibre and NCC into the UPR also did not affect the FTIR spectrum patterns of the composites; however, the peak at a wavenumber of 872 cm^{-1} was observed, associated with carbonate [30]. This peak was more apparent at the NCC content of 5 wt%, where clustering might have been occurred [27].

For the aged composites (Fig. 2b), having absorbed water until the equilibrium state, the FTIR absorbance peaks of composites did not change significantly. The notice should be focused on the wavenumbers of 3700-3200 cm^{-1}, which corresponded to the OH stretching (the absorbed water). Compared to that without NCC, the absorbance peaks between the wavenumbers of 3700 and 3200 cm^{-1} decreased up to the NCC content of 3 wt% and then increased again at the NCC content of 5 wt%. The trend of absorbance peak was in reasonable agreement with the equilibrium water uptake trend.

3.3. Tensile properties

Fig. 3a shows the typical stress-strain curves of unaged and aged bamboo fibre/NCC-modified UPR composites, selected at the NCC content of 1 wt%. For the unaged composites, the failure occurred at the peak load; however, for the aged specimens, the failure occurred after the peak load, which could be due to the gradual interfacial failure between bamboo fibre and UPR matrix.

Fig. 3b shows the tensile strength of unaged and aged bamboo fibre/NCC-modified UPR composites, while the tensile properties of unaged and aged pure UPR were shown in Table 2. The variation of data, which are the standard deviation, were indicated by vertical bars for every data set in Fig. 3. The unaged and aged tensile strengths of composites were much lower than those of the unaged and aged pure UPR, which were about 40-49 % and 65-75 %, respectively. The lower composites strength compared to that of pure UPR agreed well with the reported literature [19, 31]. For the aged pure UPR, it seemed that no significant degradation in tensile strength and elastic modulus was observed. For the aged composites, the tensile strength decreased considerably compared to the unaged composites. Hence, the decrease of the aged composites tensile strength was likely attributed by the water-induced degradation of bamboo fibre-UPR interface [6, 32]. The dif-

Table 1. The water uptake properties of pure UPR and bamboo fibre/NCC-modified UPR composites.

Materials	$D \times 10^{-3}$ (mm^2/h)	M_∞ (%)
Pure UPR	3.45 ± 0.87	1.245 ± 0.044
BF/UPR_NCC0	17.85 ± 2.42	9.032 ± 0.627
BF/UPR_NCC1	16.69 ± 0.15	8.001 ± 0.361
BF/UPR_NCC3	15.08 ± 1.08	7.565 ± 0.085
BF/UPR_NCC5	18.02 ± 2.28	9.419 ± 0.585

Table 2. Mechanical properties of unaged and aged pure UPR.

Properties	Unaged	Aged (24 days)	Aged (112 days)
Tensile strength (MPa)	51.83 ± 2.62	51.69 ± 2.62	51.94 ± 0.58
Tensile modulus (MPa)	2252 ± 93	2300 ± 79	3430 ± 55
Strain at break	0.036 ± 0.002	0.038 ± 0.007	0.034 ± 0.001
Flexural strength (MPa)	96.66 ± 5.94	84.72 ± 1.73	70.68 ± 6.82
Flexural modulus (MPa)	2343 ± 185	2445 ± 39	2177 ± 449
Displacement at peak load (mm)	10.68 ± 1.13	11.79 ± 0.44	12.73 ± 1.08
Impact strength (kJ/m^2)	0.39 ± 0.13	0.29 ± 0.00	0.33 ± 0.01

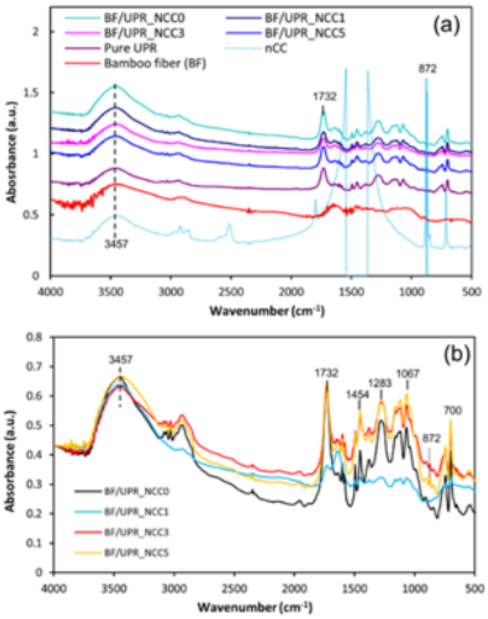

Fig. 2. FTIR spectra of bamboo fibre/NCC-modified UPR composites: (a) in dry condition, and (b) after being aged reaching the saturation level.

ference in the composite's tensile strength between ageing times of 24 and 112 days seemed not high, even the tensile strength of 112 days-aged composites slightly increased at the NCC contents of 0 and 1 wt%. It might be due the swelling of bamboo fibre that exerted the matrix and created interlocking mechanism between bamboo fibre and the matrix [33, 34]. Compared to the unaged composites, the tensile strength of the 112 days-aged composites decreased up to 47, 43, 37, and 37 % for the NCC contents of 0, 1, 3 and 5 wt%, respectively.

As seen in Fig. 3b, among the composites, the NCC addition could improve the tensile strength of the composites, but the NCC content at which the improvement was made depended on the ageing condition. For the unaged composites, by adding 1 wt% NCC, the tensile strength of the unaged composites was improved slightly (2.56 %). However, at the NCC contents of 3 and 5 wt%, the tensile strength decreased about 7.3 and 17 %, respectively. The decrease of tensile strength at high NCC content was due to the increase of viscosity resulting in the poor wetting of matrix to fibre [8, 35]. Moreover, the high NCC content increased the probability of particles agglomeration and could hinder the fibre-matrix wetting. The agglomerated particles became sites for stress concentration, promoting crack initiation [35, 36]. For the aged composites, at the ageing time of 24 days, the tensile strengths were increased by 38, 42 and 35 % after the additions of 1, 3 and 5 wt% NCC, respectively, as compared to without NCC. However, at a longer ageing time (112 days), the improvement of tensile strength was reduced to 10.4, and 8.8 at the NCC contents of 1, and 3 wt%, respectively. The improved tensile strength of aged composites was likely due to the reduced water absorption after NCC addition, which then reduced

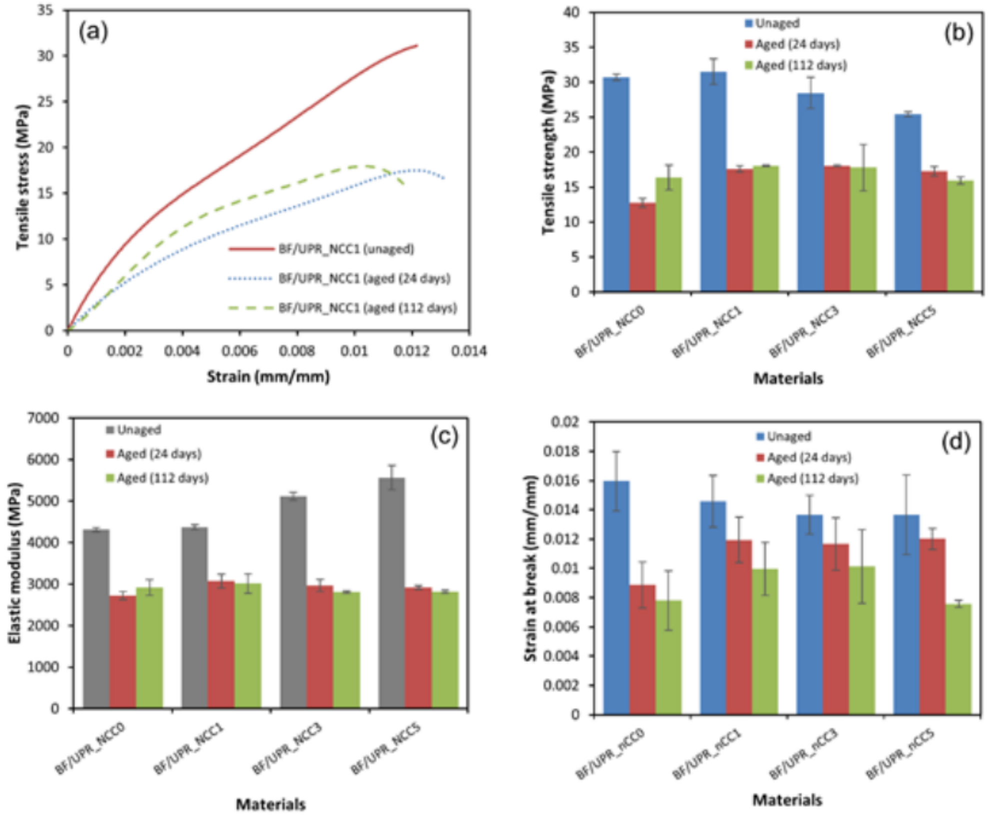

Fig. 3. (a) The typical of stress-strain curves selected at the NCC content of 1 wt%, (b) tensile strength, (c) elastic modulus, and (d) strain at break of unaged and aged bamboo fibre/NCC-modified UPR composites.

the degradation of bamboo fibre/UPR matrix interfaces [33]. Meanwhile, at the NCC content of 5 wt%, the tensile strength of 112 days-aged composites decreased by 2.6 % compared to that without NCC. In addition to the interface degradation, the decrease could be due to the agglomerated NCC particles trapped water and then reduced the matrix strength.

Fig. 3c shows the unaged and aged elastic modulus of bamboo fibre/NCC-filled UPR composites. Compared to the unaged pure UPR (Table 2), the elastic modulus of unaged composites increased by about 91.6, 94.1, 127.3, and 147.2 % for the NCC contents of 0, 1, 3 and 5 wt%, which was caused by the bamboo fibre and NCC reinforcements. However, the contributions of NCC particles at the contents of 1, 3 and 5 wt% were 1.3, 18.6 and 29 % of the total elastic modulus enhancement. Compared to the aged UPR (see Table 2), the elastic modulus of 24 and 112 days-aged pure UPR increased, but the significant increase (about 52 %) occurred for the 112 days-aged pure UPR. The increase might be due to additional cross-link in the chain structures due to water [34, 37]. For the composites, compared to the unaged composites, the elastic modulus of the 24 and 112 days-aged composites decreased significantly. It seemed that the elastic modulus of 24 and 112 days-aged composites did not differ significantly. The decreases of the elastic modulus of 112 days-aged composites were 32, 31, 45 and 49 % for the NCC contents of 0, 1, 3 and 5 wt%, respectively. The decrease was mostly determined by the degradation of bamboo fibre/matrix interfaces [32]. The degradation of bamboo fibre/matrix interfaces then reduced the effective stress transfer from matrix to bamboo fibre resulting in the decreased elastic modulus.

Among the composites, the elastic modulus of unaged composites tended to linearly increase with the increase of NCC content, following the rule of mixture. The linear increase of elastic modulus was also reported by other researchers [11, 13], which was expected as NCC has high elastic modulus compared to the matrix. Furthermore, the elastic modulus was independent on the interface strength of particle and matrix [38], so it tended to increase with the increase of NCC content. For the aged composites, the absorbed water was able to significantly decrease the elastic modulus; however, the NCC addition appeared to reduce the degradation. Compared to that without NCC, for the 24 days-aged composites, the NCC addition increased the elastic modulus by 12.8, 8.8 and 7 % for the NCC contents of 1, 3 and 5 wt%, respectively. Meanwhile, for the 112 days-aged composites, the positive contribution of NCC on the elastic modulus only occurred at the NCC content of 1 wt%. The lower contribution of NCC on the elastic modulus of the aged composites compared to those of the unaged composites might be due to the degraded bamboo fibre/matrix interfaces. The degraded bamboo fibre/matrix interfaces significantly decreased the stress transfer from matrix to fibre, so the positive effect of NCC on the elastic modulus as shown in the unaged composites diminished. This phenomenon was similar to that in a very plasticized epoxy matrix filled with inorganic particles [39].

The strain at break of the specimens is shown in Fig. 3d. The strain at break of pure UPR matrix was much higher than the composites (see Table 2). In all cases, the strain at break of the unaged composites tended to be higher than that of the aged composites. For the composites, beside matrix and fibre, the interface strength of fibre-matrix also determined the strain at break. It can be observed that for the unaged composites, the strain at break tended to decrease with the increase of NCC content, while for the aged composites, the strain at break tended to increase. For the unaged composites, the bamboo fibre-matrix interface was good, and the matrix became stiffer with the increase of NCC, so the strain at break tended to decrease. Whereas for the aged composites, the interface of fibre-matrix became poor and played the main role the strain at break.

Fig. 4 shows the typical photographs of fracture for the unaged and 24 days-aged composites. Because most the fracture surfaces within the unaged and aged composites showed a similar pattern, the photographs were only taken at the NCC content of 1 wt% as representative. It was observed that for the unaged composites (Fig. 4a) the fracture surfaces were mostly dominated by the fibre breakage, indicating that the interfacial bonding between fibres and matrix was good. However, fibre with large diameter tended to be pulled out as they have a lower aspect ratio than the smaller fibre with the same length. Meanwhile, for the aged composites (Fig. 4b), the fracture surfaces were dominated by the fibre pulled out. The absorbed water could reside in the matrix, bamboo fibre and the interface of fibre-matrix. Water at the interface could degrade the interfacial strength, and upon the loading, the bamboo fibre debonded and then pulled out from the matrix.

Fig. 5 shows the SEM micrographs of tensile fracture surfaces for the unaged and 24 days-aged composites. For the unaged composites, as seen in Figs. 5(a-c), the bamboo fibres appeared to be covered by a thin layer of unsaturated polyester matrix, which indicated good interfacial bonding. The NCC was seen as a white spot, and some were indicated by red arrows. At the NCC content of 5 wt%, a larger white spot might indicate the nanoparticles cluster. The NCC clusters could act as a stress raiser, initiating crack propagation. Hence, the clustered particles might cause the decrease of tensile strength at the NCC content of 5 wt%. While the relatively well dispersed of NCC at the content of 1 wt% increased the tensile strength of composites.

For the aged composites (Figs. 5d-f), the fibre looked cleaner than that of the unaged composites, indicating that the fibres were debonded from the matrix after absorbing water. Hence, the decrease of composite tensile strength and stiffness was mainly caused by the fibre debonding. However, introducing NCC nanoparticles decreased water absorption, which then reduced the degradation of the fibre-matrix interface due to water.

3.4. Flexural properties

Fig. 6 shows the unaged and aged flexural properties of the bamboo/NCC-modified UPR composites. The typical stress-displacement curves of unaged and aged composites (taken at the NCC content of 1 wt%) were shown in Fig. 6a. Compared to the unaged specimens, the slope of the curves of the aged specimens decreased, but the displacement at the peak load increased, indicating the stiffness degradation. Moreover, for the aged composites, the load gradually decreased after the peak load, which indicated that debonding of the fibre-matrix interface played the primary role in the failure process.

Like the tensile strength, the flexural strengths of unaged and aged bamboo fibre/NCC-modified UPR composites was lower than those of pure UPR (see Table 2), which was typical of randomly short fibre composites. The flexural strength of unaged, 24 and 112 days-aged composites were about 51-55 %, 36-42 %, and 29-41 % of the flexural strength of unaged, 24 and 112 days-aged pure UPR, respectively. After being aged, the composites degraded significantly. Compared to the unaged composites, the flexural strength of 24 days-aged composites decreased by 38,

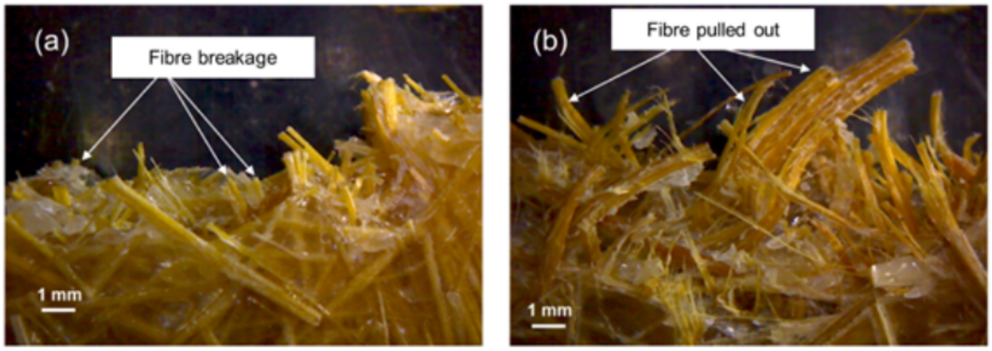

Fig. 4. Photographs of the (a) unaged and (b) 24 days-aged tensile fracture specimens, selected at the NCC content of 1 wt%.

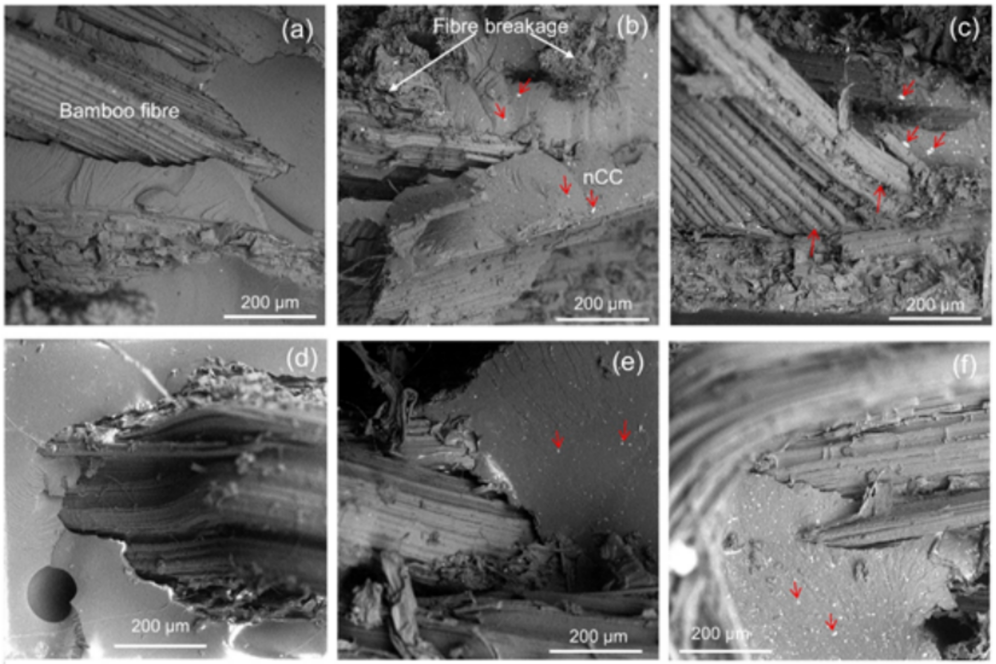

Fig. 5. (a), (b) and (c) showing the SEM micrographs of tensile fracture surfaces of bamboo fibre/NCC-modified UPR composites for the unaged specimens at the NCC content of 0, 1 and 5 wt%, respectively, while the specimens (d), (e), and (f) were for the 24 days-aged composites at the NCC content of 0, 1 and 5 wt%, respectively.

36, 35 and 29 % for the NCC contents of 0, 1, 3 and 5 wt%, respectively. Increasing ageing time to 112 days further decreased the flexural strength of composites by 58, 52, 53 and 42 % for the NCC contents of 0, 1, 3 and 5 wt%, respectively. The decrease of flexural strength of 112 days-aged composites was higher than those of the tensile strength. It indicated that in flexural load, the water- sensitivity of the composites was higher than in the tensile strength as in flexural load there were complex stresses included tensile, compressive and shear stress [34].

Compared to that without NCC, the addition of NCC improved the flexural strength of the unaged and aged

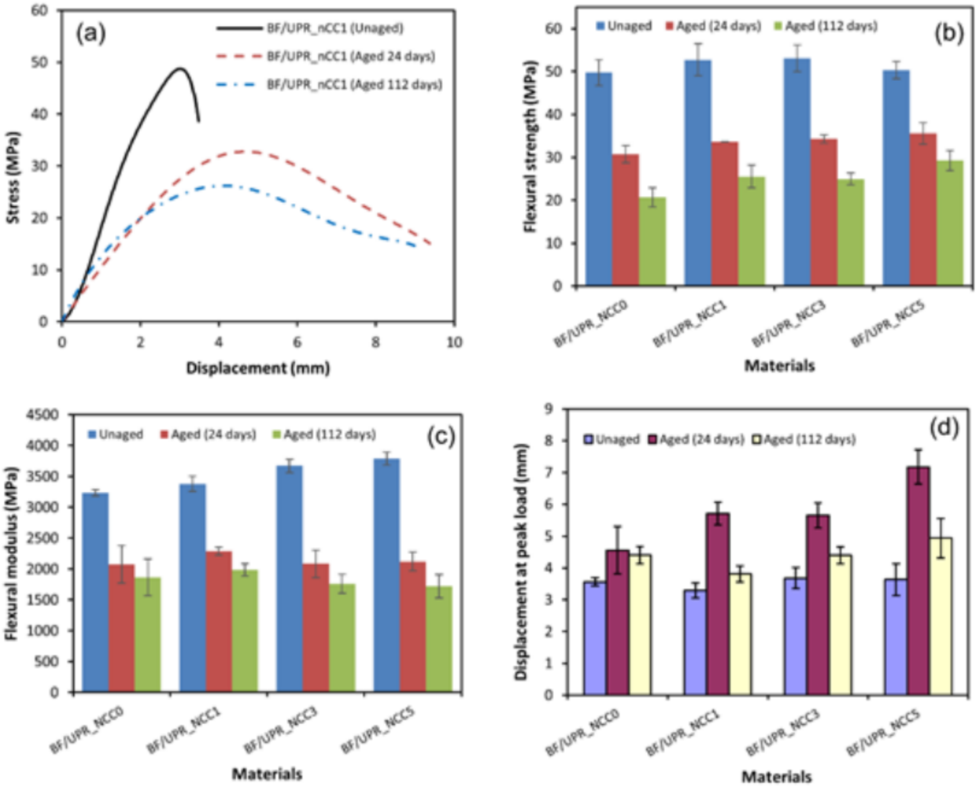

Fig. 6. (a) The typical of stress-displacement curves selected at the NCC content of 1 wt%, (b) flexural strength, (c) flexural modulus, and (d) displacement at peak load of unaged and aged bamboo fibre/NCC-modified UPR composites and pure UPR.

composites. For the unaged composites, the highest improvement occurred at the NCC content of 3 wt%, which was about 6.7 % (Fig. 6b). Meanwhile, for the aged composites, the addition of NCC reduced the degradation of the composite due to water, shown by the further improvement of the composite's flexural strengths. For the 24 days-aged composites, if compared to that without NCC, the increases of flexural strength were 9.3, 11.6 and 15.8 % for the NCC contents of 1, 3 and 5 wt%, respectively. While for the 112 days-aged composites, the flexural strength improvements were 23.4, 20.6 and 41 % for the NCC contents of 1, 3 and 5 wt%, respectively.

In contrast to the flexural strength, the flexural modulus of composites was higher than that of pure UPR (see Table 2), as expected. The flexural modulus of unaged composites also increased linearly with the increase of NCC content (Fig. 6c). As the content of bamboo fibre was constant, the increase was due to the NCC addition. The NCC in the UPR provided effective obstacles for UPR molecular chain structures to deform. The increase of dispersed NCC content (up to 3 wt%) increased the elastic modulus as the rigidity of NCC was much higher than the UPR matrix. However, for high NCC content (i.e. 5 wt%), the NCC tended to agglomerate that reduced the effective stress transfer resulting in the slight decrease of elastic modulus improvement, as seen in Figs. 3c and 6c. Similar results were also reported by Baskaran et al. [11]. The effect of NCC content on the flexural modulus of 24 and 112 days-aged composites seemed not significant. Compared to the unaged composites, the flexural modulus of 24 days-aged composites had decreased by 35, 32, 43 and 44 % for the NCC contents of 0, 1, 3 and 5 wt%, respectively. Increasing ageing time to 112 days decreased further the flexural modulus of the composites to 42, 41, 52 and 55 % for the NCC contents of 0, 1, 3 and 5 wt%, respectively. As seen in Fig. 6d, the displacement at peak for the pure UPR was much higher than the composites. For the unaged composites, the displacement at peak load due to the NCC content was not significantly different. However, ageing the composites (i.e. 24 days) increased the displacements at peak load compared to those of unaged composites. The discussion of flexural properties (the strength, elastic modulus, and the displacement at peak load) due to the NCC content and the ageing was not carried out again as the cause was quite similar to that in the tensile properties.

3.5. Impact properties

Fig. 7 shows the unaged and aged impact strengths of bamboo fibre/NCC-modified UPR composites. The impact strength of pure UPR (see Table 2) was much lower than those of the composites, so the increase of composites impact strength was mainly due to the addition of bamboo fibre. The impact strengths of aged pure UPR was lower than the unaged one, further confirming the increased stiffness of pure UPR due to water [34, 37]. For the composites, the unaged impact strength slightly improved about 14, 25 and 16 % after adding 1, 3 and 5 wt% of NCC, respectively, compared to the unfilled composites. Similarly, the slightly improved impact strength (about 13 %) was observed for both the 24 and 112 days-aged composites at the NCC content of 1-3 wt%, but then the improvement decreased to 11 % at the NCC content of 5 wt%. Compared to the unaged composites, the impact strength of 24 days-aged composites increased significantly by 92, 90, 73 and 59 % for the NCC contents of 0, 1, 3 and 5 wt%, respectively. Further ageing up to 112 days, the impact strength was slightly lower than those of 24 days-aged composites, which might be due to the decrease of matrix toughness and much lower the bamboo fibre/matrix strength. The increase and decrease of impact strength are well correlated with the failure of the composites, therefore examining the fracture surfaces as discussed below could explain the impact strength trend.

Fig. 8 shows the failure photographs of impact specimens for the unaged and 24 days-aged composites, selected at the NCC content of 1 wt%, as the representative for the unaged and aged composites. It was observed that for the unaged composites (Fig. 8a), the failure was dominated by fibre fracture, but some larger fibres were pulled out. The pulled out of larger fibre could be due to the lower aspect ratio compared to the smaller fibre, so the stress transfer capability was lower. Thin layer matrix was observed attached on the pulled out fibre. On the other hand, for the aged composites, the failure was dominated by fibre pulled out. As reported in the literature [2, 40], the impact strength was be attributed by the matrix/fibre fracture, the debonding and the pulled out fibre. Among those factors, debonding and fibre pulled out contributed more to the impact strength; therefore, the lower impact strength of the unaged composites compared to the aged composites was expected. For the aged composites, the high increase of impact strength was attributed by the massive fibre debonding and pulled out after water degraded the interface of fibre and matrix.

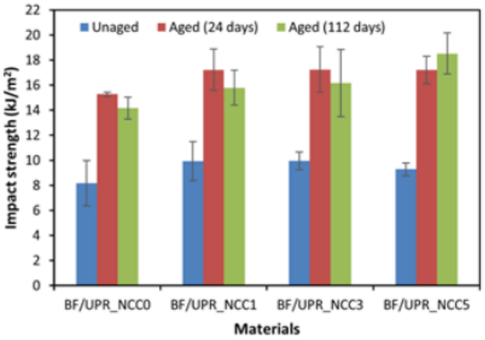

Fig. 7. The impact strength of unaged and aged pure UPR and bamboo fibre/NCC-modified UPR composites.

The SEM of the fracture surfaces for the unaged and aged composites are shown in Fig. 9. It was observed that at the NCC content of 0 wt%, the matrix fracture surfaces of the unaged (Fig. 9a) and aged (Fig. 9d) composites were smooth, indicating brittle fracture. At the NCC content of 1 wt%, the matrix fracture surfaces of unaged (Fig. 9b) and aged (Fig. 9e) were rougher than that of the unfilled matrix. It indicated that the NCC particles promoted matrix deformation and increased the matrix toughness as also reported by Sun et al. [41]. Larger particles, as seen in Fig. 9c at the content of 5 wt% might indicate agglomeration of NCC particles. For fibre reinforced composites, the impact strength was mainly determined by the debonding strength and fibre pulled out rather than the matrix toughness, so the increased matrix toughness might slightly increase the impact strength both for the unaged and aged composites.

4. Conclusion

Investigation on the durability of bamboo fibre/NCC-modified unsaturated polyester composites has been undertaken. Compared to that of unmodified UPR, adding NCC up to 3 wt% decreased both the equilibrium water uptake and the diffusion rate of composites up to 16.2 % and 15.6 %, respectively, but adding 5 wt% NCC increased

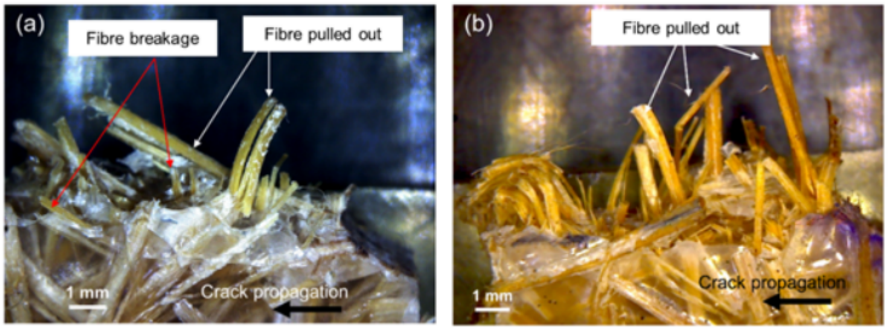

Fig. 8. Photographs of the (a) unaged and (b) aged (for 24 days) impact fracture specimens, selected at the NCC content of 1 wt%.

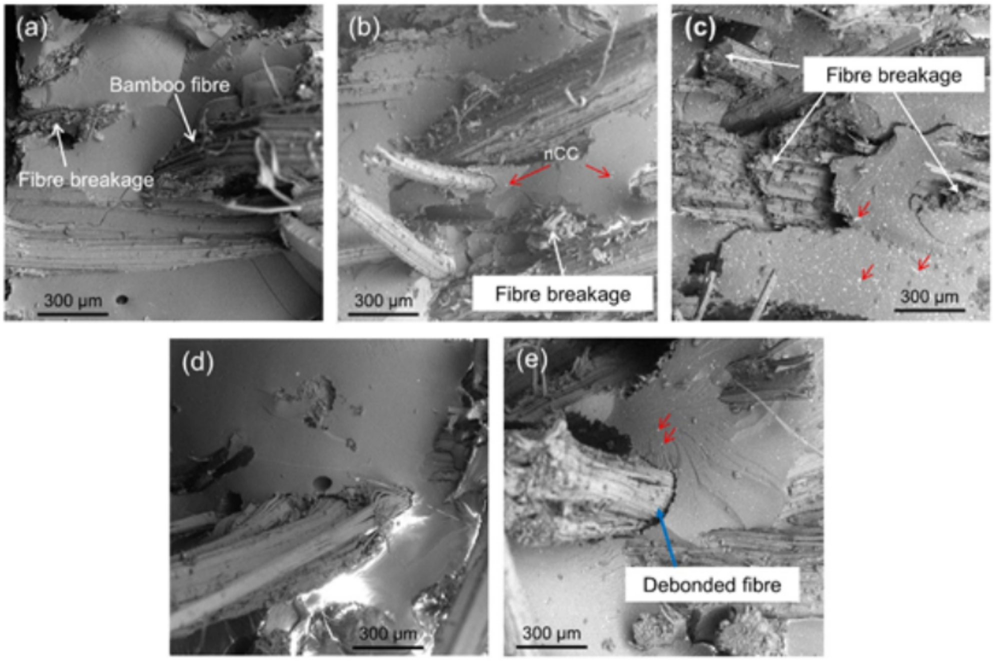

Fig. 9. SEM micrographs of tensile fracture surfaces of bamboo fibre/NCC-modified UPR composites for unaged specimens at the NCC content of (a) 0, (b) 1 an(c) 5 wt%, while (d) and (e) are for the 24 days-aged composites at the NCC content of 0, and 1 wt%, respectively.

the equilibrium water uptake and the diffusion rate to be higher than the unmodified UPR. For the unaged composites, the elastic modulus in tensile and flexural loadings tended to increase linearly with the increase of NCC; however, for the unaged tensile and flexural strengths, the best improvement occurred at the NCC content of 1 and 3 wt%, respectively. In general, adding 5 wt% of NCC did not improve the tensile and flexural strengths, but tended to decrease them. For the aged composites, NCC addition (at all the studied contents) improved the tensile and flexural strengths, but only marginally enhanced the elastic modulus at the content of 1 wt%. Meanwhile, the impact strengths of the unaged and aged bamboo fibre/UPR composites were improved at all the studied NCC contents (1-5 wt% NCC). The impact strength of aged composites increased significantly compared to the unaged composites, due to massive fibre debonding and pulled out.

5. Acknowledgements

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