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RESIDUAL STRENGTH OF EPOXY-COATED PINEAPPLE LEAF FIBER EXPOSED TO HIGH ALKALINE ENVIRONMENT

Anurak Tappakron[1], *Manote Sappakittipakorn[1], Sittisak Jamnam[1] and Piti Sukontasukkul[1]

[1]Construction and Building Materials Research Center, Faculty of Engineering,
King Mongkut's University of Technology North Bangkok, Thailand.

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ABSTRACT: Employing plant fibers to reinforce cementitious materials is feasible but not ubiquitous as fibers degrade rapidly when exposed to the alkaline-rich environment in cement composites. To alleviate such degradation and promote the use of natural fibers, processes to enhance the alkaline resistance of the fibers are of great interest. In this study, a process of coating fibers with epoxy was researched, and its efficacy was compared with two typical fiber treatment processes – boiling in water at 100°C for 60 minutes and bleaching in 10% w/w NaOH solution for 30 minutes. Pineapple leaf fiber (PALF) was the natural fiber chosen for the experiment due to its excellent mechanical properties. An untreated PALF (controlled sample) and treated PALF were examined by means of a single fiber tension test and bonding test. From the test results, the epoxy-coated PALF (EPALF) exhibited the highest average tensile loading capacity and the highest bonding force. Therefore, only EPALF was selected for further alkaline degradation testing. As the curing process of the epoxy (at room temperature) was time-consuming, it was shortened by being cured at elevated temperatures. The optimum temperature is 100 °C, which was thus used to prepare EPALF for later study, named EPALF100. To predict the fiber degradation in high alkaline environment, EPALF100 samples were submerged in 10% w/w NaOH solution at temperature of 30 °C, 50 °C, and 70 °C for 30, 60, and 90 days, and then examined the residual tensile strength. An empirical equation to estimate fiber residual strength was then proposed.

Keywords: Pineapple Leaf Fiber (PALF), Fiber Degradation, High Alkaline Environment, Epoxy Coated PALF

1. INTRODUCTION

As concrete has low fracture energy, its energy absorption is poor. Yet, it can be improved by the addition of fiber [1-4]. Reinforcing fibers are added to concrete to bridge cracks and maintain the concrete's integrity and load-bearing capacity. Fibers exist in either synthetic or natural forms. From the sustainable material point of view, natural fibers are more favored as they are more eco-friendly [5-7].

The natural fibers mostly used in cementitious composite studies are plant fibers such as hemp [8-10], jute [11], sisal [12], coconut [12], bamboo [13], etc. Their main composition is cellulose, ranging in 40–80%. The amount of cellulose plays a significant role in determining their strength. Compared to those fibers, pineapple leaf fiber (PALF) is a competitive option as it typically has a cellulose content of 70–85%. Its excellent performance has been proved in many fiber-reinforced polymer studies [14-16], but the research on employing PALF in cement composites is very limited [17].

A PALF is a fiber extracted from a pineapple leaf, which is the post-harvested pineapple residue and traditionally requires disposal. The STATISTA website reports that the annual production of pineapple worldwide in 2022 is around 29.4 million metric tons. Around 20–30% of the production consists of pineapple leaves as a byproduct [14]. Therefore, turning the discarded pineapple leaves into PALF will not only reduce agricultural waste but also provide additional extra income.

While plant fiber is a potential substitute for synthetic fiber, its practical use in concrete is rarely found. The major difficulty of using plant fiber in concrete is the fiber degradation after exposure to high alkaline environment [18]. The alkali degradation process of natural fiber consists of hydrolysis of lignin and hemicellulose, stripping of cellulose microfibrils, and deterioration of amorphous regions in cellulose chains. Numerous studies aim to mitigate the alkaline degradation of cellulosic fibers. For instance, approaches include reducing the alkalinity of cementitious matrices, treating the fiber surface, and coating the fiber with carboxylate styrene-butadiene rubber latex (XSBR). The fiber coating studies [19, 20] reported that the XSBR employed forms a protective layer to remarkably reduce the decay of jute and sisal fiber in cement-based composites. Hence, a polymeric coating has been demonstrated as an effective solution against alkaline degradation in plant fibers.

In this study, PALF was used as a core fiber, which was subjected to a key treatment with epoxy coating and was named epoxy-coated PALF (EPALF). Epoxy was chosen due to its excellent strength, stiffness, and chemical resistance. The effect of epoxy coating was assessed on the tensile and bonding load of EPALF. Furthermore, the alkaline degradation of EPALF was also evaluated.

2. RESEARCH SIGNIFICANCE

To assess the feasibility of using PALF in cement-based materials, this study examines the effect of alkaline exposure on its ultimate load capacity. To improve alkaline resistance, it was coated with epoxy, which exhibits a similar cross-linking structure to XSBR but offers superior mechanical properties. Compared to two other treatments – boiling in water and bleaching in NaOH solution, the epoxy coating yielded superior results. However, the epoxy-coated PALF still suffers from alkaline and moisture degradation. Thus, its residual strength after both types of degradation were experimentally measured and empirically modeled.

3. RESEARCH METHODOLOGY

3.1 Extraction of PALF

The pineapple leaf fibers used in this study was provided by a PALF textile manufacturer located in southern Thailand. It was delivered in the form of yarn rolls. The yarn production is briefly shown in Fig. 1 and described as follows. The company selects and collects pineapple leaves left over after pineapple harvest from all agricultural areas in Thailand. Their outer layers are first removed using a decortication machine. Afterward, they are sun-dried for 7-8 hours and extracted with a carding machine. Next, they undergo degumming and washing in 100°C water, followed by another 7-8 hours of sun-drying. Finally, they are threaded into yarn.

The physical properties of the PALF yarn are shown in Table 1. Its chemical composition based on the standard test method TAPPI T 203 and T 222 are shown in Table 2.

Table 1 Physical properties of PALF yarn

Property	Value
Specific Gravity (g/cm^3)	1.17
Tensile Strength (MPa)	162.21
Elastic Modulus (GPa)	5.59
Diameter (mm)	0.509 – 0.781
Water Absorption (%)	272 – 289

Table 2 Chemical composition of PALF yarn

Chemical Composition	wt.%
Cellulose	67.1
Hemicellulose	20.7
Lignin	10.2
Pectin and other	2

(a)

(b)

(c)

(d)

Fig.1 Production of PALF yarn; (a) pineapple leaf selection (b) decortication (c) degumming and (d) threading

3.2 Treatments of PALF Yarn

In a cement-based composite, a reinforcing PALF requires treatment to resist alkaline degradation. Inspired by the concept of polymeric coating, this study employed epoxy resin. The performance of the epoxy-resin-coated fiber was then evaluated in comparison to two other treatment methods: boiling in water and alkaline bleaching in NaOH solution. To prepare a pineapple fiber specimen for the treatments, the pineapple yarn was cut into a 200-mm long piece. Hereafter, this will be referred to as PALF.

The epoxy coating process began by submerging the PALF specimens in 70°C water for 60 minutes. Afterward, the specimens were air-dried in a laboratory environment maintained at 25 ± 1°C and 50 ± 5% RH for 24 hours before being dipped in an epoxy resin bowl. The epoxy used was Sikadur®-31 CF Normal. The epoxy-dipped PALF specimens were pultruded through a 1-mm diameter hole on a plastic plate to control their dimension. They were finally air-dried in the laboratory condition for another 24 hours. These epoxy-coated PALFs were labeled as EPALF. The magnified images of the side view (left) and cross-section (right) of PALF and EPLAF are shown in Fig. 2.

For the water boiling process, the PALF fibers were boiled in water at 100°C temperature for 60 minutes. For the alkaline bleaching, the PALF fibers were bleached in 10 wt.% NaOH solution for 30 minutes and then wash with water. Afterward, both were air-dried in the laboratory for 24 hours. The fibers treated with the boiling process were labeled BPALF, and those treated with the alkaline bleaching process were labeled NPALF.

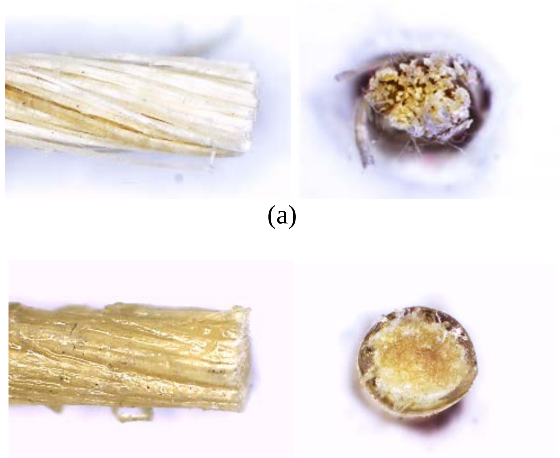
4. EXPERIMENTAL PROGRAM

4.1 Single Fiber Tension Test and Bonding Test

As the study aims to develop PALF to be a reinforcement in cement-based material, the main measurement of fiber performance was the tension test of a single fiber and the bonding test between a fiber and cement mortar. For both tests, each end of the fiber specimen was embedded in a half-dog-bone-shaped (briquette-shaped) solid specimen, which was used for holding fibers in a tension testing machine.

For the tension test, the solid specimens, as shown in Fig.3(a), were made of epoxy resin like the one for the PALF fiber coating. For the bonding test, the solid specimen, as shown in Fig.3(b), at one end was made of epoxy resin while at the other end was cement mortar. The mortar was a mix of cement, sand, and water at a ratio of 1:1.75:0.48 by weight. It yielded a compressive strength of 41 MPa (a 50-mm. cube after air-cured and wrapped with polystyrene plastic for 28 days).

To prepare a test specimen, one end of the fiber was first placed in a centerline of half of the mold. The half mold was filled with epoxy. It was cured for 24 hours before being demolded. Then, the casting process was repeated on another end of the fiber. For the tension test specimen, the mold was filled with epoxy. The bonding test specimen was filled with mortar. All specimens were covered with polystyrene and air-cured for 28 days before testing. The embedded length of fibers in the solid specimens was 25 mm. The gage length of fibers was 70 mm. The testing process utilized a universal testing machine with a 1 kN. loading capacity. For both tests, the machine was executed at the loading rate of 2 mm. per minute. The tensile load and deformation were recorded and analyzed to evaluate the performance of PALF specimens.



(a)

(b)

Fig.2 Microscopic image of the side view and cross-section of (a) PALF and (b) EPALF

(a) (b)

Fig.3 Test setup of fibers in tension testing machine (a) for ultimate tensile load test of a single fiber and (b) for bonding test between fiber and mortar

4.2 Effect of Fiber Treatments

The influence of the treatments on the ultimate tensile force and bonding force was first examined on PALF, BPALF, NPALF, and EPALF specimens. The comparison among these fiber specimens was discussed in the next section. From the test result, EPALF yields the highest ultimate load on either the tension or bonding test. Hence, only the EPALF was further evaluated.

4.3 Acceleration of Epoxy Curing

In the process of epoxy coating, the curing state was time-consuming. To accelerate the epoxy curing of EPALF, it was cured in an oven at a temperature varying from 60°C to 140°C with a 20°C interval. The oven-cured EPALF specimens were named EPALF, followed by the temperature used. For example, the EPALF cured at 60°C will be called EPALF60. The effect of the temperature on the ultimate tensile force and tack-free time of EPALF was subsequently assessed. Among the oven-cured EPALF specimens, EPALF100 was selected to be a representative of the epoxy-coated PALF due to its superior ultimate tensile load results.

4.4 Effect of Alkaline Degradation

Afterward, the influence of alkaline degradation on the tensile strength and modulus of elasticity of the oven-cured EPALF100 was examined. An alkaline exposure used to induce fiber degradation was an immersion of fiber specimens in 10% w/w NaOH solution. Apart from the alkalinity, the moisture effect was separately measured by using a plain water immersion. Both NaOH solution and water were varied at the temperature of 30°C, 50°C, and 70°C and the immersion was varied at the duration of 30, 60, and 90 days. The comparison of results from the tension test of fiber specimens before and after the exposures was employed to determine how the epoxy coating mitigates the strength decay of PALF. Moreover, based on the test results, an empirical equation to predict the residual strength of the EPALF100 in terms of the alkaline exposure duration was proposed.

5. RESULTS AND DISCUSSION

5.1 Effect of Fiber Treatment on Fiber Tensile Load and Bonding Load

From the tension test on the PALF, BPALF, NPALF, and EPALF fiber specimens, the ultimate tensile force was plotted in Fig.4. The untreated PALF exhibited the lowest average tensile loading of 25.70 N, serving as a baseline for comparison. NaOH-treated PALF (NPALF) demonstrated a marked improvement with an average loading of 50.86 N, nearly doubling the untreated fiber's strength, likely due to the removal of surface impurities and hemicellulose. Boiled PALF (BPALF) showed moderate improvement with an average loading of 43.37 N, suggesting partial surface modification. Notably, the epoxy-coated PALF (EPALF) exhibited superior performance with the highest average tensile loading of 64.26 N. This substantial increase can be attributed to the dual role of the epoxy coating. From a composite perspective, the epoxy functions as a matrix material, effectively holding and supporting the PALF. This matrix effect contributes significantly to the overall tensile strength, as the epoxy not only protects the fiber but also bears and distributes the applied load. The synergistic interaction between the PALF and the epoxy coating results in a composite system with enhanced mechanical properties. These findings suggest that epoxy coating is a highly effective method for improving both the mechanical properties and potentially the alkaline resistance of PALF, with important implications for its application in composite materials and alkaline environments.

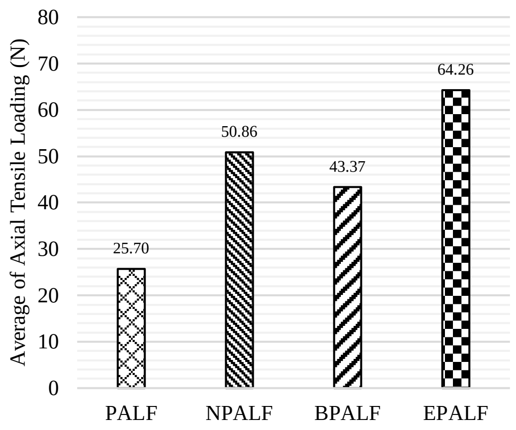


Fig.4 Averaged ultimate tensile load of untreated and treated PALF specimens

The bonding test results as shown in Fig.5 reveal significant differences in the interfacial behavior between treated pineapple leaf fibers (PALF) and cement mortar. For untreated PALF, NPALF, and BPALF, the recorded bonding forces were clearly significantly lower than their corresponding tensile strengths. This disparity indicates that these fibers experienced slippage from the cement mortar matrix before reaching their ultimate tensile capacity, suggesting inadequate interfacial adhesion. In contrast, EPALF exhibited a markedly different behavior, with a bonding force (69.38 N) close to its tensile strength. It indicates a shift in failure mode from fiber pullout to fiber breakage. The occurrence of fiber breakage in EPALF samples demonstrates that the interfacial bond strength between the epoxy-coated fibers and the cement mortar surpasses the tensile strength of the fibers themselves. This superior bonding capacity of EPALF can be attributed to several factors: enhanced mechanical interlocking due to increased surface roughness, potential chemical compatibility between the epoxy coating and cement hydration products, and the epoxy layer acting as an effective stress transfer medium.

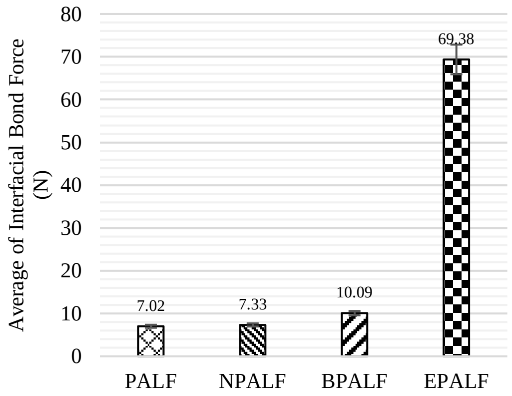


Fig.5 Averaged interfacial bond force of untreated and treated PALF specimens

5.2 Effect of Heat Curing

The tensile test results for epoxy-coated pineapple leaf fibers (EPALF) subjected to various heat curing temperatures (Fig.6) reveal significant insights into optimizing the curing process. The data shows a clear trend in tensile strength as a function of curing temperature. EPALF cured at room temperature exhibited a baseline tensile loading of 64.26 N. As the curing temperature increased, the tensile strength improved, peaking at 120.48 N for EPALF100, representing an 87.5% increase over the room-temperature cure.

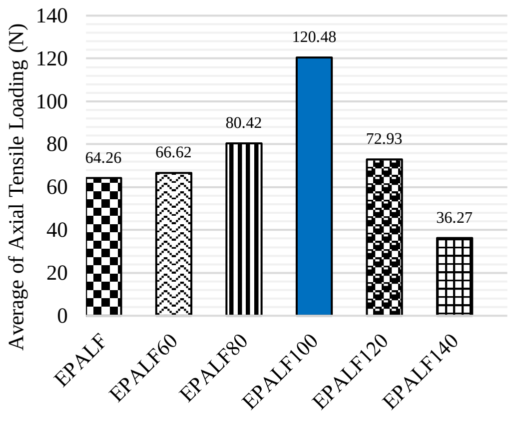


Fig.6 Averaged tensile force of EPALF after heat cured at various temperatures (the number following EPALF name is the temperature used for curing)

5.3 Effect of Heat Curing

The test results presented in Fig.7 and Fig.8 respectively reveal the influence of alkaline (NaOH) and water exposure on the tensile strength and modulus of elasticity of EPALF100 over time and at different temperatures. A general trend of decreasing mechanical properties with increasing immersion time is observed, regardless of the exposure medium or temperature [5-7]. However, the degradation rate is more severe in the NaOH solution compared to water, particularly at higher temperatures. For instance, after 90 days of immersion at 70°C, the tensile strength of EPALF100 in NaOH is approximately 20 GPa., while in water, it remains above 60 GPa. This suggests that the alkalinity of the NaOH solution accelerates the degradation process, likely due to chemical reactions between the alkaline medium and the epoxy coating or the PALF itself [12]. Temperature also plays a significant role, with higher temperatures leading to more rapid and pronounced reductions in mechanical properties over time [18]. The modulus of elasticity appears to be less affected by the exposure conditions compared to the tensile strength, indicating that the epoxy coating may be more effective in maintaining the stiffness of EPALF100 even under adverse conditions.

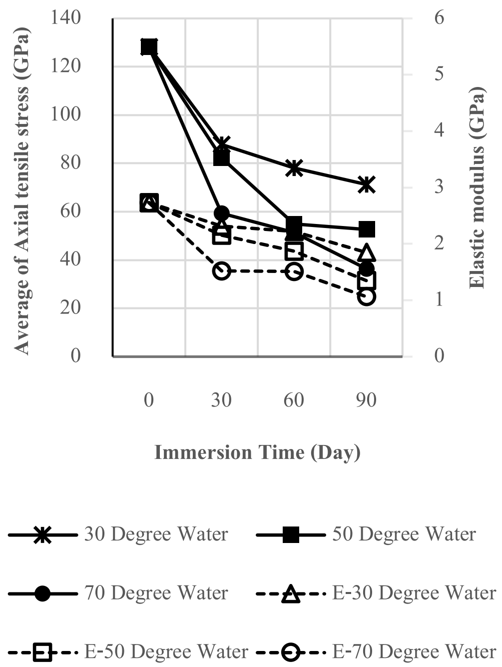


Fig.7 Average of tensile strength and elastic modulus of EPALF100 after submerged in water for 30, 60, and 90 days at 30, 50, and 70°C

The accelerated degradation in alkaline environments is likely due to the hydrolysis of the epoxy coating and the alkaline hydrolysis of the cellulose and hemicellulose in the PALF [21]. The hydroxide ions (OH-) present in the alkaline solution attack the ester linkages in the epoxy, leading to chain scission and a reduction in the coating's integrity [18]. Additionally, the OH- ions can penetrate the coating and react with the cellulose and hemicellulose in the PALF, breaking down the fiber structure and reducing its mechanical properties [7]. The effect of temperature can be attributed to the increased kinetic energy of the molecules at higher temperatures, which facilitates faster chemical reactions and accelerates the degradation process [20].

The Arrhenius equation, which relates the rate of a chemical reaction to temperature, supports this observation [22]. The less pronounced effect on the modulus of elasticity suggests that the epoxy coating may be more effective in maintaining the structural integrity of the PALF, even as the ultimate strength is compromised. This could be due to the cross-linked nature of the epoxy, which helps to distribute stresses and maintain the overall stiffness of the composite [15].

5.4 Empirical Model to Predict Strength Retention

Assuming that the sectional area of EPALF100 is constant, a percentage of its residual tensile strength (R_t) representing strength retention after degradation was calculated by the following equation.

$$\boldsymbol{R_t = \frac{P_0 - P_t}{P_0} \times 100\%} \quad (1)$$

where P_0 is the ultimate tensile strength of an undegraded EPALF100 before the degradation and P_t is the ultimate tensile strength of degraded EPALF0100 after being exposed to alkaline or moisture conditions for time t.

The calculated percentage of residual tensile strength when exposed to alkaline (NaOH) and moisture (water) environments over a period of up to 90 days was plotted respectively in Fig.9. To predict these residual strength data, an exponential decay function was applied in a regression analysis. Accordingly, an empirical equation of the residual tensile strength was proposed as,

$$\boldsymbol{R_t = e^{-[\alpha+\beta(T-30)]\sqrt{t}} * 100\%} \quad (2)$$

where $\boldsymbol{t}$ is exposure time (days), $\boldsymbol{T}$ is the temperature (°C), $\boldsymbol{\alpha}$ and $\boldsymbol{\beta}$ are constants depending on the exposure condition as follows:

for alkaline exposure in 10% w/w NaOH:
$\boldsymbol{\alpha}$ = 0.192 and $\boldsymbol{\beta}$ = 0.0008
for moisture exposure in water:
$\boldsymbol{\alpha}$ =0.064 and $\boldsymbol{\beta}$ =0.0016

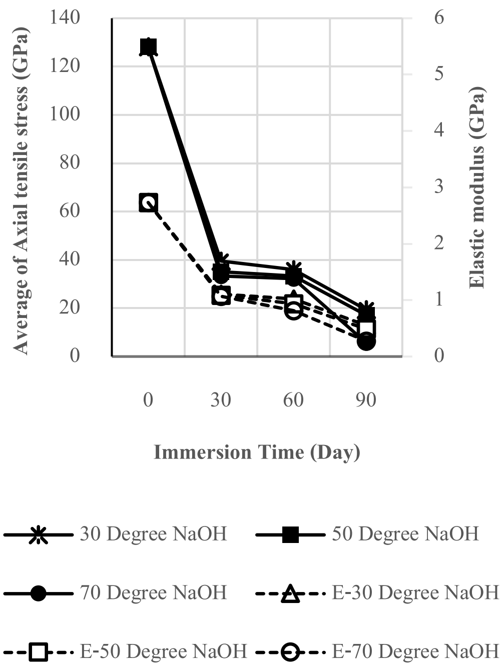
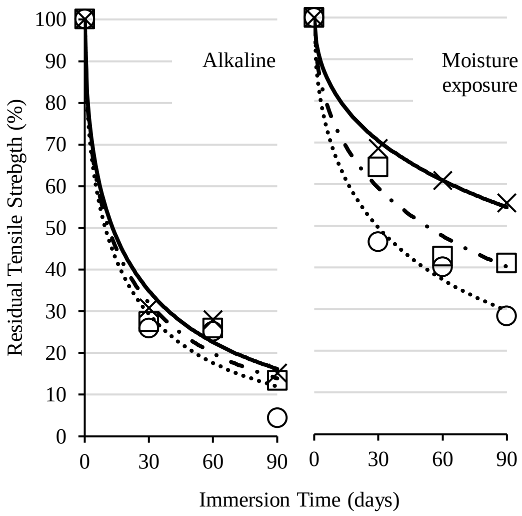


Fig.8 Average of tensile strength and elastic modulus of EPALF100 after submerged in NaOH for 30, 60, and 90 days at 30, 50, and 70°C



Temp. (°C)	Test Data	Empirical Model
30	×	————
50	□	— · — · —
70	○	………………

Fig.9 Residual tensile strength of EPALF100 after alkaline degradation

Regarding the test results, the proposed equation yields the average of standard error of regression at 3.29% and the average R-squared at 98.7%. It demonstrates a clear relationship between the exposure duration and the residual tensile strength, with higher temperatures leading to more rapid degradation. It provides a practical tool for predicting the long-term performance of EPALF100 in those environmental conditions, enabling one to estimate the residual strength of the material based on the exposure duration and temperature. However, it is important to note that these models are based on empirical data and may have limitations in terms of their applicability to other environmental conditions or exposure durations beyond the studied range.

6. CONCLUSION

The implementation of epoxy coating to alleviate alkaline degradation of pineapple leaf fiber (PALF) was experimentally assessed. From the test results, the conclusions can be drawn as follows.

- In the fiber tension test, the epoxy coating increased the ultimate load of PALF at 2.5 times of the untreated PALF. The other two treatments (boiling in water and digesting in NaOH solution) also increased the load but at lower gain at 1.7 and 2.0, respectively. In the fiber bonding test with cement mortar, the remarkable increase of bonding load was only noticed in the epoxy coating treatment.
- The curing process of epoxy coated PALF can be accelerated by heating in an oven. The heating at 100°C for 7 minutes yielded the optimum tensile load at 120.48 MPa.
- After the immersion in water and 10% NaOH solution at the temperature of 30°C, 50°C, and 70°C for the period of 90 days, the ultimate tensile load capacity of epoxy coated PALF was decreased over times. The temperature increases and the presence of alkalinity led to higher fiber degradation. Based on the test results, an empirical equation was proposed to predict the residual tensile strength of epoxy coated PALF as a function of time and temperature.

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