

Research Article

Kenaf Fibre Reinforced Polypropylene Composites: Effect of Cyclic Immersion on Tensile Properties

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This research studied the degradation of tensile properties of kenaf fibre reinforced polypropylene composites due to cyclic immersion into two different solutions, as well as comparison of the developed composites' tensile properties under continuous and cyclic immersion. Composites with 40% and 60% fibre loadings were immersed in tap water and bleach for 4 cycles. Each cycle consisted of 3 days of immersion and 4 days of conditioning in room temperature (28°C and 55% humidity). The tensile strength and modulus of composites were affected by fibre composition, type of liquid of immersion, and number of cycles. The number of immersion cycles and conditioning caused degradation to tensile strength and modulus of kenaf fibre reinforced polypropylene composites. Continuous and cyclic immersion in bleach caused tensile strength of the composites to differ significantly whereas, for tensile modulus, the difference was insignificant in any immersion and fibre loadings. However, continuous immersion in the bleach reduced the tensile strength of composites more compared to cyclic immersion. These preliminary results suggest further evaluation of the suitability of kenaf fibre reinforced polypropylene composites for potential bathroom application where the composites will be exposed to water/liquid in cyclic manner due to discontinuous usage of bathroom.

1. Introduction

Polymeric composites with various natural reinforcements (e.g., natural fibres, woods, and hybrid fibres) received significant attention in numerous applications including automotive and construction industries [1, 2]. However, the application of natural fibre composites usually intended for interior application; one of the main reasons is degradation of mechanical properties of natural fibre composites in the presence of water. Recent studies [3–8] have reported that the effect of water uptake in natural fiber composites limits their outdoor applications. In general, there are three ways to understand the term “water absorption” such as water diffusing (1) directly into the matrix, (2) through interphase matrix/reinforcements, and (3) by imperfections, like pores and cracks. Once it enters the polymers, it can

exist in several ways: as bound water, characterized by strong interaction of the molecule with matrix and free water, present in capillaries and micro cavities within the polymer [9]. When moisture diffuses into composites, it degrades fiber-matrix interfacial bonding, lowers the glass transition temperature, swells, plasticizes, hydrolyzes, and sometimes microcracks the matrix [10]. Although water absorption can cause degradation of mechanical properties, redrying process can help the materials in regaining certain percentages of their mechanical properties [11, 12]. Generally, the moisture diffusion in composite depends on factors such as fiber weight fraction, void volume, viscosity of matrix, chemical treatment of natural fiber, humidity, and temperature [13].

Haniffah et al. reported that, under cyclic immersion, longer duration of immersion and shorter drying exhibit the greatest amount of water retained in kenaf/PP

composites [14]. The tensile properties were also affected by increasing temperature of water immersion. Generally, it is reported that the tensile properties decreased with increasing immersion time [15–18]. Moreover, the mechanical degradation increased as the number of cyclic immersions increased. However, lesser degree of degradation occurred in composites with fibres modified using coupling agents [19, 20]. Similar studies on natural fiber reinforced polymeric composites have shown that the sensitivity of certain mechanical and thermal properties to moisture uptake can be significantly reduced by using coupling agents and fiber surface treatments [21, 22].

Besides soap, bleach is one of the common chemicals used in bathroom causing physical (e.g., colour) and mechanical changes to the composites that affect the perception of durability of the composites. Kenaf/PP composites experienced degradation by the chemical reagent, bleach [23]. The degradation of kenaf PP composites in bleach solution mostly occurs on kenaf fibres because PP was resistant to bleach attack [24]. The aim of this study is to compare the effect of bleach and water under cyclic immersion on tensile properties of kenaf/polypropylene (PP) composites for potential bathroom application.

2. Materials and Methods

2.1. Materials. Kenaf plant (variety V36) used in this study was supplied by National Kenaf and Tobacco Board's (NKTB), Malaysia. Polypropylene, grade TitanPro 6331, was purchased from Titan Chemicals Corp. Sdn., Bhd Johor, Malaysia. Liquids for immersion used in this treatment were tap water (pH 7.67) and domestic bleach (Clorox Regular, Malaysia) with active ingredient, for example, sodium hypochlorite 5.25%. Prior to treatment, bleach solution (pH 11.64) was prepared with 200 mL of bleach diluted in 1 L of tap water (16.17% v/v).

2.2. Measurement of Liquid Content. Liquid content in the composite was measured using the following:

$$\text{Liquid content (\%)} = \left(\frac{\text{Weight (g)} - \text{Initial Weight (g)}}{\text{Initial Weight (g)}} \right) \times 100\%. \quad (1)$$

Negative value might occur if composites are immersed in bleach especially for longer cycle. This indicates that the bleach causes some parts of composites to dissolve and to be lost in the liquid during conditioning.

2.3. Composite Preparation. The processing of kenaf/PP composites was done at Institute of Tropical Forestry and Forest Product (INTROP), Universiti Putra Malaysia (UPM). The composites were prepared with 40% and 60% fibre loadings using corotating extruder (Micromac, Micromagna, Perak, Malaysia). Then, the produced pellets were injected into tensile specimen mould using JSW 85-ton injection moulding machine model J85EM 110H using injection pressure of 78.72 MPa and injection speed of 48 mm/s. The temperatures

of injection moulding barrel were 195 and 210°C for 40 and 60% fibre loading, respectively. The specimen was prepared following type I of ASTM D638 standard and four replicas were tested for each parameter of treatment.

2.4. Methods of Treatment. All samples underwent 4 cycles of immersion and conditioning with each cycle consisting of 3 days of immersion and 4 days of conditioning. After the fourth day of conditioning, the specimens were tested using universal testing machine (Instron). The other set of specimens underwent immersion for 12 days and was conditioned for 4 days in order to evaluate the tensile properties of the composites under cyclic and continuous immersion. Three determined factors in this study were composition (40% and 60% fibre loadings), liquid of immersion (water and bleach), and cycle of immersion (the 0th, the 1st, the 2nd, the 3rd, and the 4th cycle). Two statistical analysis methods, ANOVA *F*-Test and Fisher's least significant difference (LSD), were used to determine the significant difference between experimental results.

3. Results and Discussion

3.1. Liquid Content in Composite. Liquid content in composite was the general term used in this study to describe the amount of liquid in composite at certain time regardless of whether composite was in immersion or conditioning stage. Liquid absorption was the amount of liquid absorbed by composite during immersion stage, while liquid retention was amount of liquid retained by composites during conditioning stage. The results of sorption pattern are presented in Figure 1 with all factors included in the graph. Composites were immersed in liquid and conditioned at room temperature for 4 cycles. During the first cycle of immersion, two distinguishable patterns were observed for composites with 40 and 60% fibre loading. Liquid of immersion (bleach and water) caused the patterns of liquid content to differentiate from each other with increase in cycle. The difference in pattern of liquid content was more apparent at the end of last cycle in composites with 60% fibre loading than composites with 40% fibre loading. It is clear that, even after the conditioning, some of the water absorbed by the fibers could not be removed from the composites and the amount of liquid retained inside the composite also increased with increase in immersion cycle. On the other hand, the amount of liquid retained by the composite during bleach immersion was lower as compared to water. Moreover, the highest uptake of the solution was nearly constant for immersion cycles 3 and 4.

3.2. Effect of Cyclic Immersion on Tensile Strength. Tensile strength of the composite influenced by combined effect of material composition, liquid of immersion, cycle of immersion, and conditioning is shown in Figure 2. It is seen that the fibre loading clearly influenced tensile properties of the composites; increase in fibre loading caused reduction in tensile strength at all immersion cycle. The liquid absorbed caused fibre to become more ductile during pull-out due to Poisson's effect; that is, fibre length increased with decrease in diameter.

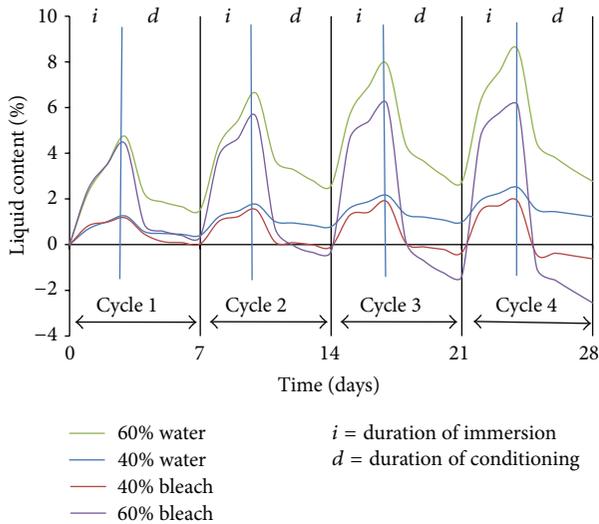


FIGURE 1: Pattern of liquid content during immersion and conditioning of kenaf/PP composites.

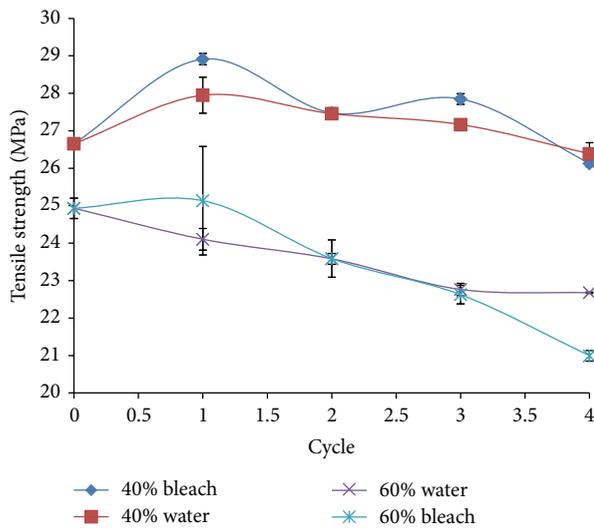


FIGURE 2: Tensile strength of PP composites with 40% and 60% of kenaf fibres loadings due to repeated immersion and drying.

The liquid also filled the gap between fibre and matrix and acted as lubricant during fibre pull-out. Residual stress might be produced due to uneven cooling or compaction during injection moulding. The early penetration of liquid, during dimensional expansion of fibre, might cause the fibre to release the residual stress exerted on it. However, the liquid could act as a lubricant when the amount of penetrated liquid increased.

3.3. Statistical Analysis on Tensile Strength. Statistical analysis is presented in Table 1. *F*-Test on three factors has been carried out to confirm the significance difference on tensile strength caused by compositions (composites with 40% and 60% fibre loadings), types of immersion liquid immersed into (water and bleach), and cycles (cycle 0, cycle 1, cycle 2,

cycle 3, and cycle 4). The differences between parameters of composition and cycle were also significant. Thus, it was confirmed that fibre loading in the composites affected tensile strength of composites.

Although the *F*-Test indicated that the difference between cycles was significant, the actual difference between cycles was shown by Fisher's least significant difference (LSD) method as presented in Table 2. The significant increase in tensile strength during the first cycle of immersion was verified by statistical analysis (Table 2). Then, tensile strength dropped to a value similar to cycle 0 and cycle 3. Possibly the residual stress, originating from processing of the composites, was released at early stage of immersion and thus improved tensile strength of the composite. However, as the immersions repeated, the liquid seeped between matrix and fibre and reduced the efficiency of loads transferred from matrix to the fibre that resulted in lower tensile strength of the composites.

In the analysis of liquid, *F*-Test in Table 3 shows that liquids of immersion (bleach and water) did not give significant difference regarding tensile strength of the composite, but there was significant interaction in *F*-Test between liquid and cycle (noted by Liquid * Cycle in Table 1). Therefore, each cycle underwent *F*-Test to isolate the interaction effect. *F*-Tests results showed that liquid of immersion significantly affected tensile strength of composites only on cycle 4. LSD analysis as shown in Table 3 reconfirmed significant difference between water and bleach in cycle 4. Therefore, the degradation in cycles 2 to 3 was observed to be higher due to the effect of water whereas only on the fourth cycle the bleach affected tensile properties of kenaf/PP composites.

3.4. Effect of Repeated Immersion on Tensile Modulus. Modulus (stiffness) is another basic property of composites; the primary intention of filler incorporation is usually to increase the stiffness of the resultant material. However, moisture absorption could significantly reduce the modulus of the composites. Clear differences amongst the effects of fibre loading, immersion cycles, and liquid of immersion on tensile modulus of the composites are observed (Figure 3). As the immersion cycle was increased, a reduction in modulus with both liquid immersions was observed. However, this reduction in tensile modulus was drastic and significantly higher in composites with 60% fibre loading as compared to 40% fibre loading. The moisture uptake, due to the immersion process, changes the structure and properties of fibers and matrix and the interface between them [18]. Moreover, as fiber content was increased, water absorption was expected to be increased [25, 26]. High fiber content in the composite leads to more water penetration into the interface through the micro cracks induced by swelling of fibers creating swelling stresses that led to composites failure [18]. The extent of strength and stiffness loss depends upon aging time and temperature also. Thwe and Liao [27] reported on bamboo-fiber composites that both the tensile strength and modulus decreased after aging in water at 25 and 75°C for prolonged period.

The composites immersed in bleach solution had lower modulus compared to water immersed composite samples.

TABLE 1: ANOVA for tensile strength of kenaf/PP composite with 40% and 60% fibre loadings immersed in water and bleach.

Source	DF	Sum of squares	Mean square	F value	Pr > F
Composition	1	242.32	242.328	384.28	<0.0001
Liquid	1	0.7029	0.70291	1.11	0.2953
Composition * Liquid	1	0.0183	0.01831	0.03	0.8653
Cycle	4	64.168	16.0421	25.44	<0.0001
Composition * Cycle	4	17.019	4.25486	6.75	0.0001
Liquid * Cycle	4	11.245	2.81119	4.46	0.0032
Composition * Liquid * Cycle	4	0.5062	0.12656	0.20	0.9370
Error	60	37.836	0.63061		
Corrected total	79	373.82			

TABLE 2: Means comparison among cycles for tensile strength.

t grouping	Mean	N	Cycle
A	26.4623	16	Cycle 0
B	25.7923	16	Cycle 1
B C	25.4007	16	Cycle 2
C	24.9134	16	Cycle 3
D	23.7962	16	Cycle 4

TABLE 3: Compilation of mean comparison among liquid at each cycle.

Cycle	Liquid		Significant (Y/N)
	Water (MPa)	Bleach (MPa)	
0	25.7923	25.7923	N
1	26.026	26.8985	N
2	25.5191	25.2822	N
3	24.9653	24.8616	N
4	24.5308	23.0616	Y

This is perhaps due to degradation of the natural fibers by bleach solution, which might have caused the fiber-matrix interfacial debonding. The potential for fiber/matrix debonding and microcracking at the interface during wet/dry cycling of composites is dependent upon (among other factors) the dimensional stability of the fiber reinforcement in response to moisture fluctuations. Mohr et al. [28] showed that composites produced with fibers, which were wetted and dried prior to the mixing, exhibited superior dimensional stability compared to composites produced with fibers, which had never been dried. The bleached (i.e., low-lignin) composites exhibited accelerated progression of fiber mineralization as compared to unbleached fibers. Unbleached composites exhibited greater toughness, particularly for low numbers of wet/dry cycles. Without exposure, unbleached fiber composites exhibit greater flexural properties than bleached fiber composites.

3.5. Statistical Analysis on Tensile Modulus. Statistical analysis with *F*-Test in Table 4 showed that the difference between parameters of composition, liquid, and cycle was significant.

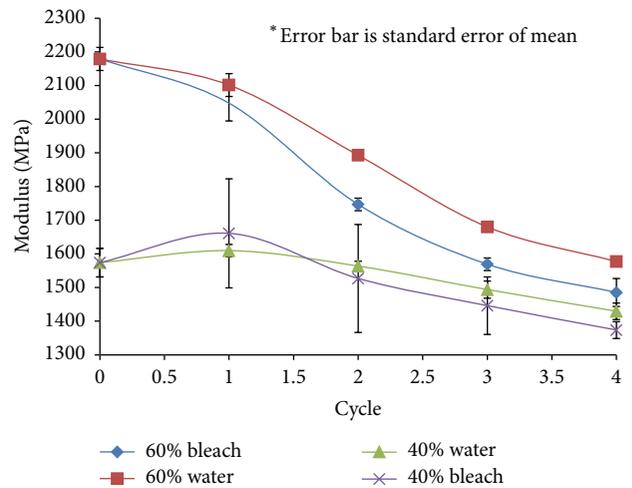


FIGURE 3: Tensile modulus of kenaf/PP composites with 40% and 60% fibre loadings due to repeated immersion and drying.

Since all the factors are significant, the interaction between the factors does not influence the results of ANOVA *F*-Test.

Both composition and liquid of immersion had only two parameters; the significant difference in Table 4 referred to composites with 40% and 60% fibre loadings and also water and bleach, respectively. On the other hand, cycle had five parameters and LSD test was used to compare which parameters were different. Table 5 showed that cycles 2, 3, and 4 differ from all cycles but cycles 0 and 1 do not differ from each other. This proved that significant degradation only started on cycle 2 onward.

The experiment confirmed that cycle of immersion, composition, and liquid of immersion had effects on tensile modulus of kenaf/PP composites. However, the cycle of immersion only affected tensile modulus of the composites on the second cycle onward.

3.6. Difference in Tensile Strength between Cyclic and Continuous Immersion. Result of tensile strength of composites with 40 and 60% fibre loading immersed in water and bleach is presented in Figure 4. Both types of immersion had caused degradation in composites with 60% fibre loading immersed in bleach. Tensile strength of composite with 40%

TABLE 4: ANOVA for tensile modulus of 40 and 60% fibre loading kenaf/PP composites immersed in water and bleach.

Source	DF	Sum of squares	Mean square	F value	Pr > F
Composition	1	2057626	2057626	535.48	<0.0001
Liquid	1	48306	48306	12.57	0.0008
Composition * Liquid	1	19701	19701	5.13	0.0272
Cycle	4	2117627	529407	137.77	<0.0001
Composition * Cycle	4	644814	161203	41.95	<0.0001
Liquid * Cycle	4	32139	8035	2.09	0.0931
Composition * Liquid * Cycle	4	8649	2162	0.56	0.6906
Error	60	230554	3843		
Corrected total	79	5159415			

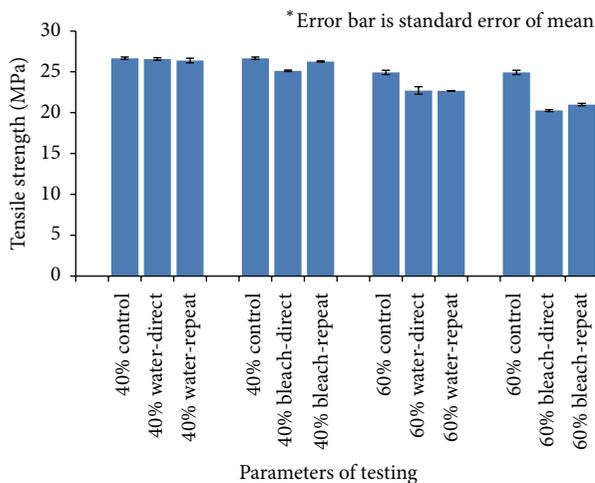


FIGURE 4: Difference of tensile strength between control, continuous (direct), and cyclic (repeat) immersion for 40 and 60% fibre loading of kenaf/PP composites.

TABLE 5: Means comparison between cycles for tensile modulus.

t grouping	Mean	N	Cycle
A	1876.53	16	Cycle 0
A	1854.93	16	Cycle 1
B	1682.55	16	Cycle 2
C	1547.28	16	Cycle 3
D	1466.32	16	Cycle 4

fibre loading immersed in water under continuous and cyclic immersion did not significantly differ. Tensile strength of composite with 40% fibre loading immersed in bleach was degraded under continuous immersion but, under cyclic immersion, tensile strength remains almost the same as composite without any immersion. It might be possible that conditioning process in cyclic immersion reduced the activity of bleach on the composites.

3.7. Differences in Tensile Modulus between Cyclic and Continuous Immersion. Both continuous and cyclic immersions had reduced tensile modulus of composites (refer to Figure 5). Composites with 40% fibre loading had loss around

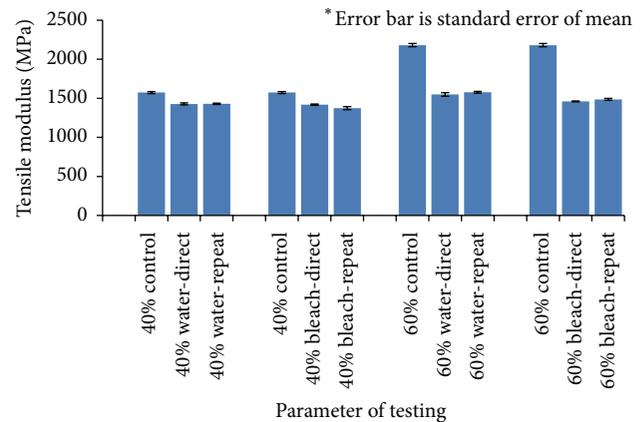


FIGURE 5: Difference of tensile modulus between control, continuous (direct), and cyclic (repeat) immersion for 40% and 60% fibre loading of kenaf/PP composite.

100 MPa of tensile modulus while composites with 60% fibre loading had loss around 600 MPa of tensile modulus due to 12 days of immersion. Furthermore, composites under continuous and cyclic immersion had similar value of tensile modulus. Therefore, the reduction of tensile modulus is not influenced by type of immersions.

4. Conclusions

Both continuous and cyclic immersions in bleach affected the tensile strength of the kenaf fibre reinforced polypropylene composites. Increased number of cycles of immersion and conditioning caused degradation of tensile strength and modulus. However, continuous immersion in the bleach reduced the tensile strength of composites more compared to cyclic immersion.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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