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Mechanical and Thermal Characterization of Alkali Treated Kenaf Fibers

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Abstract. Research on bio composite for automotive and aerospace application has been extensive with the advancement of natural fiber yarn and woven technology. Malaysia has marked kenaf as its main crop commodity by 2020. Surface modification of natural fibers is one of the significant areas in current biocomposite research. Alkali treatment removes certain amount of lignin, hemicellulose, and wax on the surface of fiber, besides depolymerizing cellulose structure and increasing percentage of crystallinity. Surface modification with NaOH of 3%, 6% and 9% concentration with various lengths of immersion time was conducted. The effect of alkali treatment on the mechanical strength and thermal degradation of kenaf fibre were investigated by means of fiber bundle tensile test and thermogravimetric analyser (TGA). Alkali treatment strongly modifies the thermal behaviour of the fibers, being particularly effective in the removal of noncellulosic matter. In addition, the mechanical properties of kenaf fibers revealed higher tensile strength for NaOH treated fibers.

1. Introduction

Manufacturing high performance composites from natural fibres is actively pursued by researchers across the globe. Automotive and aerospace industry has started on natural fiber composite since 1999, with Kenaf fibers as seat backs, packaging trays, door inserts, load floors and pillars, in vehicle interiors [1]. Boeing developed sidewall panel for a 737 aircraft made of flax-thermoset composite in 2013[2]. BMW i3 with its lightweight design produced side panel trim and instrument panel made of hemp in 2013, and Kenaf in random mat form in 2014 [3].

Kenaf is the name given to fiber from the bast of stems of plants of the genus *Hibiscus*, family Malvaceae and species *H. cannabinus* L. In Malaysia the government is heavily promoting the development of kenaf, as the next major industrial crop for the country in line with its policy in developing new sources of economic growth. Kenaf has a short gestation period of only four months and is a high carbon dioxide absorbent [4]. Kenaf growth does not need pest control and absorbs chemical and heavy metals from the soil. It also has a wider range of adaptation to climates and soils than any other fiber plant in commercial production [4].

Not unlike other natural fibers, kenaf as plant fiber offers the undisputable advantage of renewability for which production requires little energy and involves carbon dioxide absorption whilst returning oxygen to the environment. It also has the advantage of low hazard manufacturing processes and less abrasion to processing equipment, besides involving much less emission of toxic fumes when subjected to heat as compared to current synthetic materials [5]. However, the main disadvantages of



natural fiber as composite reinforcements are the poor compatibility between fiber and matrix, flammability and high moisture sorption [6].

This leads to an important area in natural fiber composites which is pre-treatment of fibers. Alkaline treatment commonly involves sodium hydroxyde (NaOH) reacting with the hydroxyl group of the natural fibers, removing hemicellulose, minimal amount of lignin, wax and oil that surround the external surface of the fiber, leading to increase in surface roughness [7]. This facilitates mechanical interlocking which leads to an improvement in fiber-matrix adhesion. [8]. In addition, alkalisation caused fiber fibrillation i.e. axial splitting of the elementary fibers, leading to a decrease in fiber diameter, increasing the aspect ratio and the effective surface area available for wetting by a matrix in composite [9]. High moisture content in fibers is attributed to presence of hemicellulose, and removal of hemicellulose by alkalization has been reported to reduce moisture absorption in natural fiber [10]. Alkalization also improved the strength of individual fibers. Removal of hemicelluloses cause the inter fibrillar regions of the fiber to becomes less dense and rigid, hence fibrils are more capable of rearranging themselves along the direction of tensile deformation, promoting even load distribution in the fibers and reduces stress concentration [11]. Edeerozey et al immersed kenaf in NaOH solution with different concentrations (3%, 6% and 9% NaOH) for 3 hours at room temperature and fiber bundle tensile test showed NaOH 6% giving the highest strength[12]. Changes in chemical constituents of natural fiber due to alkalization improved the strength and moisture content of fiber and composites fabricated from.

Natural fibers are combustible in nature. When exposed to fire or high intensity heat source, polymer matrix and fibers will thermally decompose to yield flammable and non-flammable volatile gases [13]. The released flammable volatile gas will react with surrounding oxygen and form the final combustion product accompanied by heat release, which will continue the combustion cycle [14]. Literature have reported that variations in the flammability of different plant fibers can be attributed in part to the chemical composition of the fibers [15]. Cellulose is known to have high flammability due to its high crystallinity, whereas hemicellulose and lignin improves fire resistance; hemicellulose produced non-combustible gas whilst lignin produced char that reduces the amount of flammable volatiles released, slowing the chain branching reactions in the combustion cycle [15]. Hence alkalization affects flammability with the change in chemical composition it caused. Thermal degradation condition is crucial in the understanding of flammability properties of material. Thermal stability is one of the means by which natural fiber develop their inherent flame resistant [28].

Thermal degradation of natural fibers has always been a crucial factor for the development of natural fiber composites. The majority of natural fibers have low degradation temperatures of below 200°C due to its cellulosic nature [16]. Alkalization has been reported to increase thermal stability as reported by Guduri et al. on flax fibers [17]. Liu et al. reported that the temperature at the maximum rate of decomposition obtained through weight percentage and derivative weight percentage for the Indian grass fibres increased with the increase in alkali concentration, thereby resulting in the improvement of the thermal stability of the fibre after alkali treatment [18]. However there has been contradicting results, as reported by Puglia et al. who indicated that thermal stability of the alkali-treated flax fibres (Phormium tenax) decreased slightly as shown by the earlier degradation temperature as compared to untreated flax fibres [19]. Similar results have been reported by Zhu et al. based on their experiments on also flax fibres [20]. Research in thermal degradation of alkali treated kenaf composite is scarce, even more so on the kenaf fiber itself.

This research work aims to study the effect of alkali treatment on the thermal degradation and mechanical strength of kenaf fibers. This preliminary study will lead to an in depth understanding of fire and mechanical behaviour of alkali treated kenaf composites, with regards to the main aim of this research which is the manufacture-ability, durability and other functional properties of kenaf composites.

2. Samples preparation and experimental procedure

2.1. Materials selection

Kenaf fibre with enzyme retting extraction grade was obtained from Dynamic Agrofarm Sdn Bhd (Pahang, Malaysia) at the harvesting age of 4 months. The extraction method was conducted by first spraying the kenaf stem with pectinase-rich enzyme and letting it rest for 24 hours. This was followed by manually peeling the kenaf bast from the core, and soaking it in a tank of water with the enzyme for 3 days, and finally dried under sunlight for 24 hours. The kenaf fibers were cut into an average length of 100 mm.

2.2. Alkali treatment

Sodium hydroxide (analytical grade) pellets purchased from Merck were prepared of 3%, 6% and 9% concentration. The kenaf fibers of 100 mm length were separated into ten groups of 20 grams each and each group (except one) was immersed into the three different NaOH concentration for duration of 1, 3 and 24 hours. The fibers were then washed with running water mixed with 0.1% acetic acid until a neutral pH is achieved, dried in a hot air oven at 60°C for a period of 24 hours to remove the excess moisture.

Table 1. Designation of kenaf fibers.

Designation	Treatment	Soaking Time
Raw	-	-
NaOH3% 1hr	NaOH 3%	1 hour
NaOH3% 3hr	NaOH 3%	3 hours
NaOH3% 24hr	NaOH 3%	24 hours
NaOH6% 1hr	NaOH 6%	1 hour
NaOH6% 3hr	NaOH 6%	3 hours
NaOH6% 24hr	NaOH 6%	24 hours
NaOH9% 1hr	NaOH 9%	1 hour
NaOH9% 3hr	NaOH 9%	3 hours
NaOH9% 24hr	NaOH 9%	24 hours

2.3. Fiber bundle tensile test

Kenaf fiber exists in fiber bundles. In accordance with ASTM D 3822, the tensile properties of fiber were determined with Shimadzu Ez-L10NX fiber tensile strength testing machine. The sample was set at fiber gauge length of 10 mm at the crosshead speed of 5 mm/min, and tested as in figure 4. For each of the ten sets of sample as in table 1, 20 fiber bundles with diameter less than 60 μm were chosen and tested individually to determine the average fiber bundle strength. Prior to the testing, average fiber bundle diameter were obtained from biological microscope.

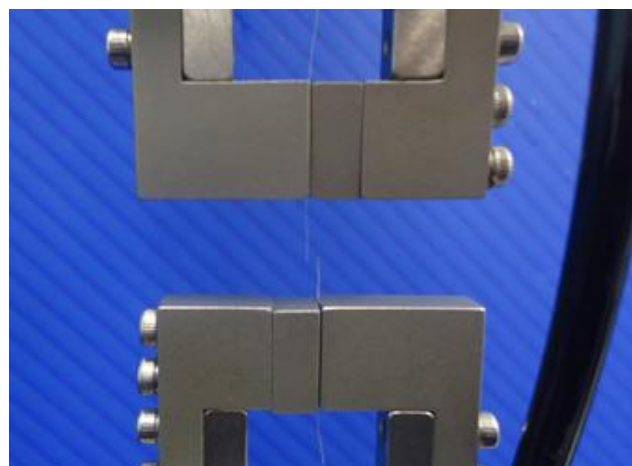


Figure 1. Fiber tensile strength testing machine.

2.4. Thermogravimetric analysis (TGA)

Thermogravimetric analysis is a thermal analysis techniques used to measure the mass change, thermal decomposition and thermal stability of material. Thermal gravimetric analysis was conducted using TGA Q500 machine. Samples weighing approximately 5 mg were subjected to pyrolysis in nitrogen environment to a maximum temperature of 650 °C at a heating rate of 20 °C/min. The weight loss was recorded in response to increasing temperature, with final residue yield on set of degradation temperature and number of degradation steps reported.

3. Results and discussion

3.1. Physical properties

Kenaf fibers exist in a fiber bundle form. This is observed when the fiber was partially pulled apart. Figure 1 shows a visual representation and the magnified image of untreated kenaf fiber in its fiber bundle form obtained from a microbiological microscope of 100 μm magnification. From literature, micro scale fiber bundle exists in 50-100 μm diameter [21]. A single kenaf fiber can only be obtained by breaking down the kenaf fiber bundles into elementary fibers, with diameter of 10-20 μm [21].

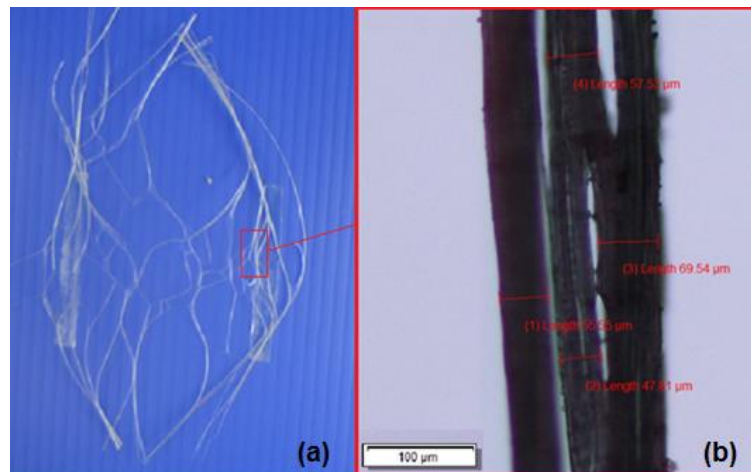


Figure 2. (a) kenaf fiber bundle (b) kenaf fiber bundle magnified at 100x.

For the purpose of studying the fiber bundle strength, only those with diameter between 40 to 65 μm were chosen. The diameter of each fiber was measured at three different random locations to obtain the average value, as shown in figure 2. Fibers of more than 65 μm were observed to be observably intertwined and were separated for tensile strength test.

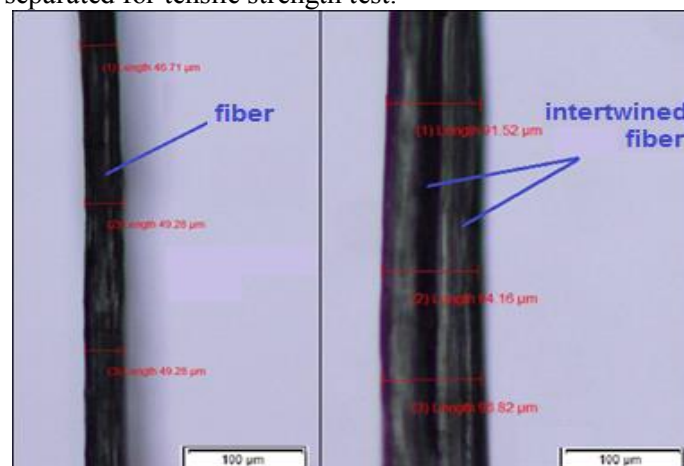


Figure 3. Magnified fiber bundle image.

It was observed that as the alkali concentration increases, the fiber bundles were easier to separate, demonstrating partial removal of hemicellulose and lignin as binding agent had occurred at the lower NaOH concentration and a more complete removal had occurred at the higher NaOH concentration. Fibers treated with higher concentration also showed a more severe physical change. Figure 3 shows a comparison of raw and kenaf fiber treated with concentration of 3%, 6% and 9% treated for 24 hours. During alkalization, almost all of hemicellulose and a certain amount of lignin, wax and oil covering the external surface of the fiber cell wall are removed, thus causing the fibers to appear dry and bristly. Hemicellulose is a semi crystalline polymer of glucose that acts as a binding agent to the cellulose strand. Hemicellulose is covered with lignin, an amorphous polymer which also acts as a binder. Removal of hemicellulose and partial removal of lignin caused the kenaf fibers to loose most of its binding agent [22]. Figure 3 showed a comparison of physical change shown by kenaf fibers soaked for duration of 24 hours. Samples soaked at the lesser time showed the same rough and bristly condition but a lesser severity.



Figure 4. Untreated and NaOH treated kenaf fibers (24 hours).

3.2. Fiber bundle strength test

The overall results for kenaf fibers treated for soaking time of 1 hour, 3 hours and 24 hours were summarized in table 2. All three soaking time shows a similar pattern, which is an increase of average maximum tensile strength of the fibers with increasing concentration of NaOH, up to 6% NaOH. However, NaOH concentration of 9% show a dramatic drop of strength, with value recorded even lower than that of the untreated fibers. Figure 5 shows tensile strength of untreated and treated kenaf fibers of various NaOH concentrations treated for 1 hour.

Table 2. Average tensile strength (MPa).

Soaking Time	Raw	NaOH 3%	NaOH 6%	NaOH 9%
0 hour	330.42	-	-	-
1 hour	-	358.89	407.72	309.84
3 hours	-	367.39	412.29	297.65
24 hours	-	330.25	386.37	267.54

This increased tensile strength with the increase of NaOH concentration may be explained by the improved uniformity of cellulose structure after the removal of the amorphous and semi-crystalline hemicellulose and lignin [18]. The aligned fibrils promotes an even load distribution in the fibers, and a higher crystallinity index leads to a better packing of cellulose chains [11]. The dramatic drop of strength could be the result of the damage of fiber cell wall caused by high concentration of NaOH, as a similar scenario was observed by P. Methacanon [10]. As reported by Mwaikambo and Ansell [23],

a very high concentration of NaOH would certainly damage the fiber and consequently reduce the tensile strength of the fiber.

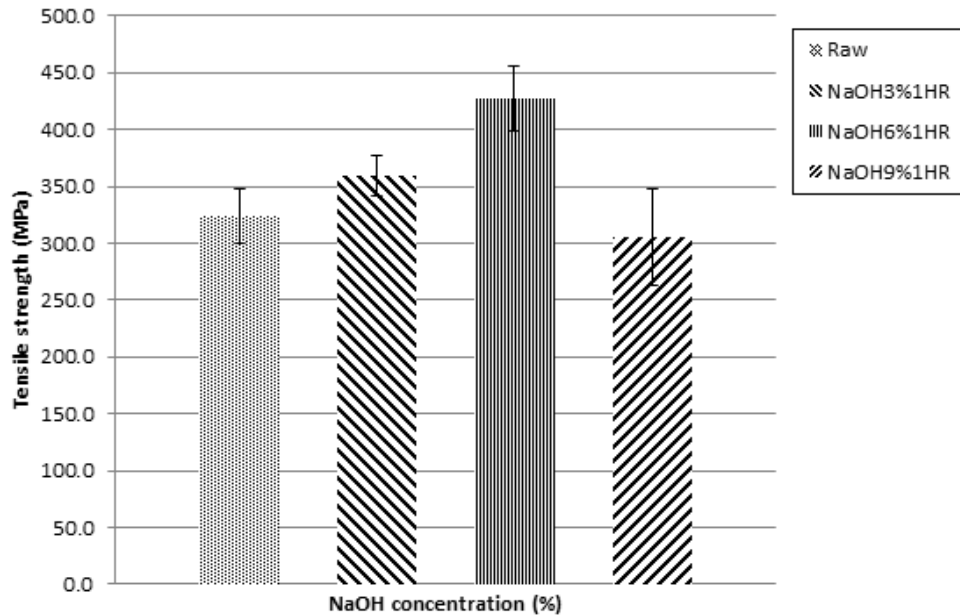


Figure 5. Tensile strength of untreated and treated kenaf fibers of various NaOH concentrations treated for 1 hour.

3.3. Thermogravimetric analysis (TGA)

Results from the TGA run is as shown in figure 6, and a clearer presentation of degradation is as presented in the derivative thermogravimetric graph (DTG) in figure 7. Peaks represent the rate of weight degradation (%/min). Untreated kenaf fibers started to lose weight earlier than the other samples as shown by the curve below 100°C. This is attributed to the higher moisture content on untreated fibers due to the presence of hemicellulose.

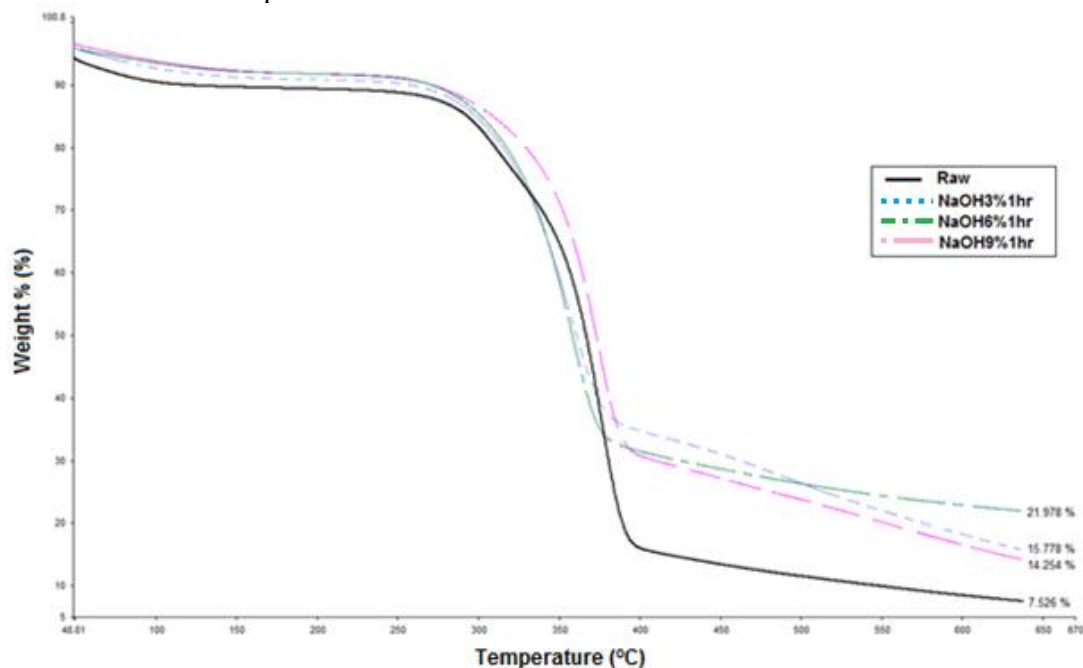


Figure 6. TGA of raw and treated kenaf fibers.

Initial result on thermal stability is characterized by the temperature at which 15% weight loss occurred, referred to as $T_{15\%}$. The $T_{15\%}$ for raw kenaf is 292.78°C as compared to 308.57°C for kenaf treated with NaOH 9%, with NaOH 3% and NaOH 6% having $T_{15\%}$ values in between. The higher temperature for 15% weight loss is an early indication of thermal stability of treated as compared to the untreated kenaf fibers [24].

Table 3. Data analysis from TGA and DTG.

Designation	Treatment	$T_{15\%}$ (°C)	Peak height (maximum weight loss)(wt%/min)	Char residue at 636.4°C (%)	Total weight decomposition (%)
Raw	NIL	292.78	30.653	7.526	65.52
NaOH3% 1hr	NaOH 3%	297.82	18.195	15.778	48.18
NaOH6% 1hr	NaOH 6%	301.3	22.275	21.978	59.98
NaOH9% 1hr	NaOH 9%	308.57	25.724	14.254	59.55

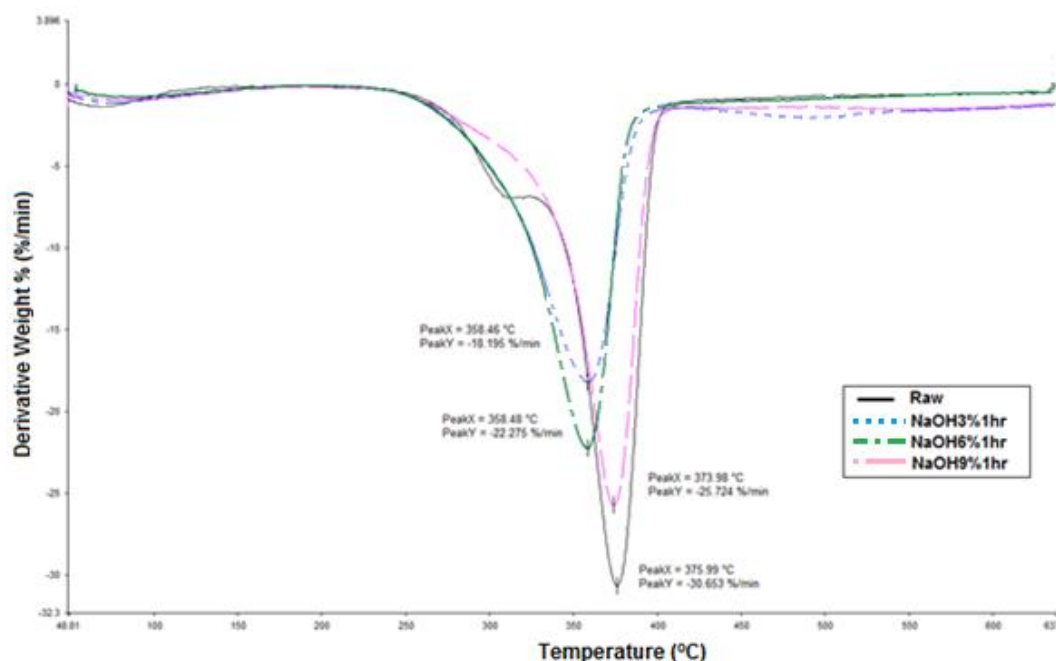


Figure 7. DTG of raw and treated kenaf fibers.

DTG curve showed that whilst treated kenaf fiber exhibits single step degradation, raw kenaf fibers displayed twostep degradation, with the first peak occurring at 270 °C. In this temperature range, degradation is associated with hemicellulose and some parts of lignin. Treated kenaf fibers did not experience loss of weight at 270°C, which is explained by the removal of hemicellulose during alkalization [25]. DTG graph of fibers treated with 9% NaOH showed a complete removal of hemicellulose as no peak is observed.

The second stage is associated with degradation of cellulose and lignin. Peaks at the range of 320 to 375°C exhibits decomposition of cellulose, and a wider range of up to 420°C is for the decomposition of lignin [23]. DTG curve showed NaOH treated fibers (3% and 6%) with an earlier degradation temperature than untreated fibers. This indication of thermal instability might be due to the removal of fiber cell walls, which reduce the thermal protection of the treated fibres. The exposed cellulose fibrils experienced direct heat without any layering from the cell wall [26]. Cellulose will degrade through a process called pyrolysis, forming levoglucosan which will decompose to produce flammable volatiles [14]. Another factor causing thermal instability in treated fiber is the crystallinity. For cellulosic fibers

in particular, higher levels of crystallinity results in higher levels of levoglucosan during pyrolysis and consequently increased flammability [15]. This is in accordance to reports of increased flammability for fibers with higher cellulose content [15].

The peaks on the DTG represent the maximum rate of weight loss. Treated fibers have a much lesser rate of degradation due to the removal of most cementing components. By treating the fibers with NaOH 3%, the maximum rate of weight loss is reduced by almost half from 30.653 wt%/min to 18.196 wt%/min, indicating thermal stability [27]. Similarly, total decomposition weight, as obtained from the area under the DTG curve, also showed a lesser decomposition prior to pre-treatment of fibers. However, the degradation rate is observed to increase as NaOH concentration used increased. This may be due to the removal of lignin, which leads to lesser char in the fibers to retard degradation.

4. Conclusion

Kenaf fibers were treated at various concentrations of sodium hydroxide and soaking time to study the effect of alkali treatment on the physical, mechanical and thermal properties of kenaf fibers. From this study, it can be concluded that:

1. Tensile properties of fiber bundle increase with NaOH treatment of up to NaOH concentration of 6%. Removal of impurities improves the uniformity of the fibers resulting in an increase in strength. However a high concentration of 9% will degrade the strength of fiber cell walls. Duration of soaking of 1 to 3 hours is sufficient for a good treatment.
2. Absence of hemicellulose in treated fibers is observed by the one step degradation instead of a two-step degradation depicted by raw fibers. The peak at 270°C correlates with the decomposition temperature of hemicellulose.
3. Treated fiber showed a slower rate of degradation and reduced total weight decomposition due to the absence of hemicellulose and lignin.
4. Exposed cellulose structures and diminishing fiber cell wall of alkali treated fibers accelerated degradation process. High crystallinity of treated fiber also caused formation of levoglucosan, decreasing thermal stability in fibers. This showed that fire resistance of fibres may not be improved with alkali treatment. Therefore, it is suggested that further study on the effect of fire retardant on thermal stability of the kenaf fibres is conducted.

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