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### Effect of aluminum trihydroxide on flame retardancy and dynamic mechanical and tensile properties of kenaf/poly(lactic acid) green composites

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## Effect of aluminum trihydroxide on flame retardancy and dynamic mechanical and tensile properties of kenaf/poly(lactic acid) green composites

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In this work, the effect of aluminum trihydroxide (ATH) on the flame retardancy and dynamic mechanical and tensile properties of kenaf/poly(lactic acid) green composites was explored. Prior to composite fabrication, poly(lactic acid) (PLA) pellets filled with ATH powder at different loadings (0, 10, 20, 30, and 50 wt.%) were processed by using a twin-screw extrusion technique. The green composites were fabricated using the pellets and chopped kenaf fibers by a compression molding method. The kenaf fibers were used as primary reinforcement, and the contents were fixed to 40 wt.% of the total, and the average fiber length was 3 mm long. It was noticeable that the flame retardancy of kenaf/PLA green composite with ATH was improved by 66%, and the storage modulus and tensile modulus were enhanced by 136 and 59%, respectively, compared with the kenaf/PLA counterpart without ATH. It was concluded that the ATH incorporated into PLA by extrusion technique played a significant role not only as flame-retardant but also as secondary reinforcement of kenaf/PLA green composites.

**Keywords:** kenaf; poly(lactic acid); green composite; aluminum trihydroxide; flame retardancy; mechanical properties

### 1. Introduction

During the last decade, natural fiber-reinforced green composites or biocomposites have been utilized to replace conventional glass fiber-reinforced polymer composites due to diverse advantages of natural fibers consisting of green composites. The main advantages of green composites over glass fiber composites are environmentally friendliness, low density, low cost, natural abundant renewable sources of natural fibers, acceptable mechanical properties, biodegradability, and carbon dioxide sequestration in nature.[1–3] An excellent review on research efforts on biocomposites reinforced with natural fibers performed during the last decade was published.[4]

In recent years, a biodegradable polymer poly(lactic acid) (hereinafter referred to as PLA) has increasingly attractive attentions as alternative of a petroleum-based polypropylene (PP).[5,6] PLA is the most promising biodegradable polymer resin with good mechanical properties and processability. Therefore, natural fiber-reinforced PLA matrix composites have been extensively studied in green composite or biocomposite systems, [7–10] which are now being utilized in automobile parts, building materials, and other

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industrial applications. In fact, a large number of papers on green composites or biocomposites have been studied on the surface modification or treatment in order to increase the interfacial properties between the cellulose-based natural fibers and the polymer matrix in a composite system and ultimately to improve their mechanical properties.[11–13]

Since PLA has low flame and fire resistances, as similarly in general purpose thermoplastic polymer resins, its uses are restricted.[14] As a consequence, enhancing flame retardancy of green composites is of great importance. However, a small number of papers have been reported on the flame retardancy of green materials.[15–17] Although chemical approach between a flame-retardant and a polymer material may be useful, the simplest and easiest way to impart flame retardancy to polymeric materials is, in general, physically incorporating flame-retardants into a targeting polymer resin.

There are a variety of commercially available flame-retardants such as halogen-containing, silicon-containing, melamine-containing, metal-based flame-retardants.[18] Among them, metal hydroxides, such as aluminum trihydroxide (ATH) and magnesium hydroxide (MH), provide an attractive alternative to halogen-containing flame-retardants for polymers, which are harmful in health during incineration and also corrosive for metals.[18] Metal hydroxides can also be easily incorporated into a polymer resin physically or mechanically, are commonly used inorganic flame-retardants with PP, which can be replaced to PLA in many applications due to its comparable features. Metal hydroxide flame-retardants have low toxicity, corrosion, and smoke during processing and burning.[18] Among aluminum-based flame-retardants, ATH is most widely utilized.[19] They can decompose endothermically and release water during burning. When heated, these water molecules can help to suppress a flame by reducing the heat and temperature of targeting materials [18] It has been known that very high loadings of metal hydroxides are usually introduced to reach a acceptable level of flame retardancy.[18,20]

Three test methods, such as Limiting Oxygen Index (LOI) test, UL 94 vertical burning test, and cone calorimetry, are well known to test the flame retardancy of plastics and composites. Among them, the LOI test is simple, fast, and inexpensive. This method is applied to quantify a material's resistance to ignition according to ASTM D2863 or ISO 4589. The more the oxygen required for the test, the higher the flame retardancy of the material, resulting in the higher LOI value. Since air contains 21% oxygen, plastics with the LOI value lower than 21 are classified as combustible materials, whereas plastics with the LOI greater than 21 are often classified as self-extinguishing materials.[21]

Many papers on kenaf/PLA composites have been reported during the past years. [9,22–24] However, reports on the flame retardancy of kenaf/PLA green composites have been scarcely found. Consequently, the objective of the present study is to investigate the effect of nonhalogen-free inorganic ATH on the flame retardancy, thermal stability, and the dynamic mechanical and tensile properties of kenaf/PLA green composites in terms of LOI, thermal stability, storage modulus,  $\tan \delta$ , and tensile properties. The composites were fabricated by means of compression molding with PLA pellets with different ATH loadings, which were obtained through twin-screw extrusion process.

## 2. Experimental

### 2.1. Materials

Kenaf was cultivated in Bangladesh and kenaf fibers used in this work were supplied from Jute Institute of Bangladesh. Aluminum trihydroxide (ATH,  $\text{Al}(\text{OH})_3$ ) used as retardant was purchased from Chemizone Inc., Suwon, Korea. PLA pellets (Grade 2003D)

was purchased from NatureWorks® (USA). The PLA has the melting point of 155 °C and the glass transition temperature of 55 °C. The density is 1.24 g/cm<sup>3</sup>. Kenaf fibers, and PLA pellets were sufficiently dried at 70 °C for 24 h in a convection oven prior to use.

## 2.2. Extrusion and compression molding processes

A twin-screw extruder was used to compound ATH powder and PLA pellets. Prior to compounding, The PLA and ATH mixed with different ratios in a polyethylene bag were mixed manually at different ratios and then dried at 70 °C for 8 h. The ATH contents in the PLA were 10, 20, 30, and 50% by weight. The twin-screw extruder (BT-30-S2-421, LG Machine Co., Korea) has modular intermeshing co-rotating screws with the diameter of 30 mm and the  $L/D$  ratio of 42 (Figure 1). The screw speed was 130 rpm, and the feeding rate was 5.5 kg/h. Three kneading disk block configurations were used to efficiently mix and compound PLA pellets with ATH. The barrel temperature was in the range of 130–180 °C. Table 1 indicates the barrel temperatures varying according to the barrel zone during extrusion process. The PLA/ATH extrudate was cooled down with cold water and uniformly cut using a pelletizer. The pellets were dried in a convection oven prior to fabrication of kenaf/PLA green composites.

Kenaf/PLA green composites were fabricated by a compression-molding method (GE-122S, Kukje Scien Co., Korea) using PLA/ATH pellets and chopped kenaf fibers. The ‘as-supplied’ kenaf fibers were chopped to about 3 mm in average by using a natural fiber chopper (DHS-28, Dae Heung Machinery Co., Korea). The fiber contents were 40% by weight. The ATH loadings containing in the composites were 0, 10, 20, 30, and 50 wt.% of PLA. Compared with the total weight of the composite, the ATH loadings were 0, 6, 12, 18, and 30 wt.%. The molding temperature was 180 °C, and the molding pressure was elevated stepwise: 500  $\psi$  for 30 min, 1000  $\psi$  for 5 min, and then 1500  $\psi$  for 5 min. The mold was naturally cool down to ambient temperature. The dimensions of the green composites obtained were 150 × 100 × 3.5 mm. Each composite plaque was cut with a band saw (BAS 250G, ELECTRA BECKUME, Germany) for analysis and test.

## 2.3. Characterization

Scanning electron microscopy (SEM, JSM 6380, JEOL, Japan) was performed to observe the fracture surface topography of PLA plaques and kenaf/PLA green composites without and with ATH. Each sample was fractured in a liquid nitrogen bath for microscopic observations. All the samples were uniformly coated with platinum for 3 min by a sputtering method.

Thermogravimetric analysis (TGA Q500, TA Instruments, USA) was used to examine the effect of ATH on the thermal stability of kenaf/PLA green composites. The

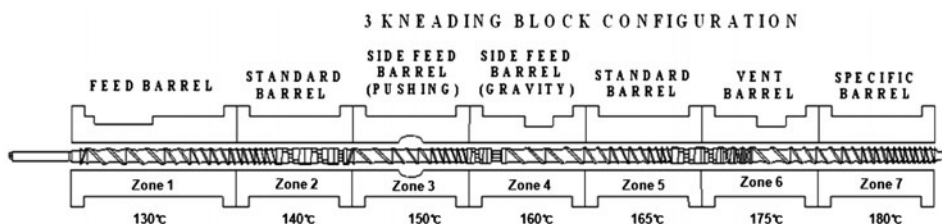


Figure 1. Screw configuration of a twin-screw extruder with modular intermeshing corotating screws used in this work ( $\phi = 30$ ).

Table 1. Variations of the barrel temperature used during extrusion processing.

Hopper	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Die
110 °C	130 °C	140 °C	150 °C	160 °C	165 °C	175 °C	180 °C	180 °C

heating rate was 20 °C/min. Each measurement was carried out up to 500 °C with purging N<sub>2</sub> gas. A can-type alumina sample pan with the depth of 10 mm was used.

The flame retardancy of kenaf/PLA green composites was evaluated by measuring the LOI. The LOI test was conducted at ambient temperature using a vertical-type oxygen index tester (Oxygen Index, FTT Co., UK) according to ISO 4589. During the test, a slow stream of oxygen/nitrogen mixture gas was supplied at the bottom of a tube in the apparatus, and a small candle-like flame was applied to the top of the specimen to ignite it.[21] The LOI was determined by measuring the minimum oxygen concentration in nitrogen that will support the combustion of each composite specimen for at least 3 min or for the loss of 5 cm in length of each specimen. The specimen dimensions were 80 mm long and 10 mm wide. A set of five specimens was used to obtain the LOI value of each composite. The LOI value of each sample was determined by the following relationship, described in the ISO 4589.

$$\text{LOI} = C_f + k \cdot d$$

where  $C_f$  is the final value of the oxygen concentration, in volume percent to one decimal place, used in the series of measurements performed in accordance with the ISO 4589,  $d$  is the interval (i.e. 0.2), in volume percent to at least one decimal place between oxygen concentration levels used and controlled, and  $k$  is a factor being determined according to responses (pass or fail) for the last five measurements. Here,  $k$  values can be referred to from ISO 4589.

Dynamic mechanical analysis (DMA, Q800, TA Instruments, USA) was carried out to measure the storage modulus and  $\tan \delta$  of kenaf/PLA green composites without and with ATH. A dual cantilever mode was applied throughout the DMA measurement. Each DMA measurement was performed from 30 to 150 °C with the heating rate of 3 °C/min. The oscillation amplitude was 0.2 mm, and the sinusoidally oscillating frequency was 1 Hz. The dimensions of each specimen were 60 × 12 × 3.2 mm.

Tensile tests were performed using a universal testing machine (UTM, AG-50kNX, Shimadzu, Japan) according to DIN 53 445. The grip distance was 115 mm, and the gage length was 100 mm. The crosshead speed was 5 mm/min. The load cell of 50 kN was used. The specimen dimensions were 150 × 15 × 3.5 mm. The average tensile strength and modulus values were obtained from 10 specimens of each composite sample.

### 3. Results and discussion

#### 3.1. Fractured surfaces

Figure 2 displays the fractured surfaces of PLA plaques without and with various ATH loadings observed by a scanning electron microscope. It was clearly seen that there were ATH particles in the range of 1–10 μm in size embedded in the PLA matrix, and the particles were distributed increasingly with increasing the ATH loading from 10 wt.% (B) to 50 wt.% (E). The surfaces became rougher with increasing the ATH. Also, it was likely that the interfacial contacts between the ATH particles and the surrounding PLA matrix

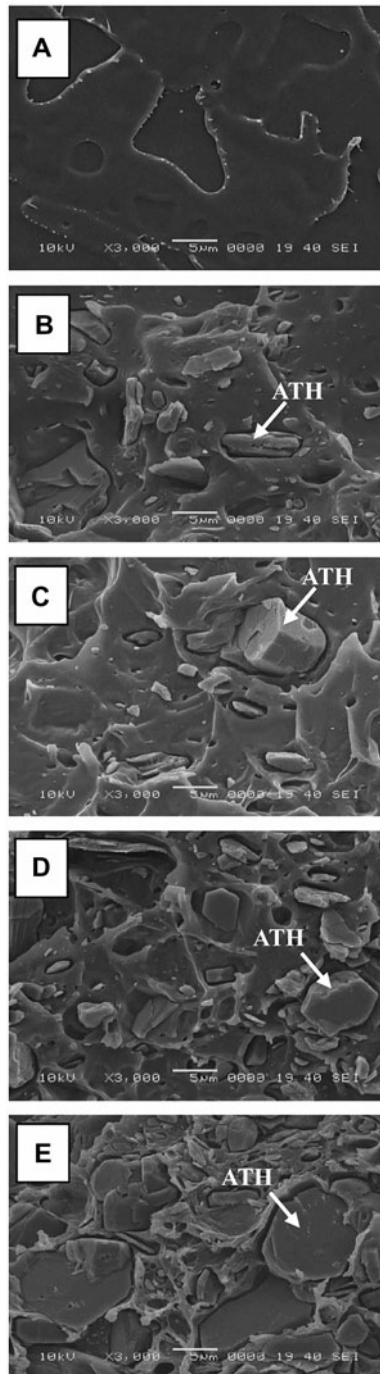


Figure 2. SEM images of the fractured surfaces of PLA films made from pellets without (A) and with various ATH loadings. (B) 10 kenaf/PLA, (C) 20 kenaf/PLA, (D) 30 kenaf/PLA, (E) 50 kenaf/PLA pellets.



were good. It was also found that at 50 wt.% some ATH particles were touched each other. Neat PLA plaque (A) without ATH exhibited more or less brittle fracture pattern.

Figure 3 shows the fractured surfaces of kenaf/PLA green composites without and with various ATH loadings. It seemed from Figure 3(A)–(D) that the adhesion between the kenaf fibers and the PLA matrix including the ATH was relatively good due to sufficient amount of PLA matrix surrounding the individual kenaf fibers. The PLA contents in the composites were between 42 wt.% (Figure 3(D)) and 60 wt.% (Figure 3 (A)) of the total. The contents of PLA, ATH, and kenaf fibers in the composite in Figure 2(E) were 30, 30, and 40 wt.%, respectively. Some agglomerated region in the matrix due to the presence of relatively large amount of ATH in the PLA matrix was found, as similarly observed from the PLA/ATH sample without kenaf in Figure 2(E). It was expected that such the agglomeration may result in microstructural defects of the matrix in the composites.

### 3.2. Thermal stability

Figure 4 exhibits the thermal stability of kenaf/PLA green composites with different ATH loadings. The ATH loading hereinafter indicates the ATH contents against the PLA matrix in the composite. From the TGA curves, the thermogravimetric behavior below about 300 °C was similarly seen. In the range of 270 to 350 °C, the weight loss occurred in the green composites with ATH was greater than that in the composite without ATH. This was because the ATH in the PLA matrix began to decompose near 270 °C.[25] It was also reported that ATH has a decomposition temperature in the range of 180–340 °C, with a peak at about 320 °C.[18] On the contrary, the thermal stability of the composites with ATH above about 350 °C was significantly higher than that without ATH, leaving more residual weight behind with increasing the ATH content. In particular, at 500 °C, the residual weight (char yield) of the composite without ATH was less than 2% but that with 50 wt.% ATH was higher than 30%. The residual weight was increased with increasing the ATH content in the PLA matrix. The finally residual weights at 500 °C were somewhat higher than the amount of ATH incorporated in the composite. Considering of some possible residual weight from PLA itself, this TGA result is in good agreement with the ATH contents in the composites. It may also be said that ATH played a role in absorbing surrounding heat and cooling down the resin matrix by releasing water molecules. [6,7] From this result, it may be expected that ATH can contribute to enhancing the flame retardancy of green composite, reflecting the increased char yield at high temperatures.

### 3.3. Limiting oxygen index

Table 3 compares the LOI values among kenaf/PLA green composites without and with ATH at various loadings. The LOI value of the composite without ATH was 23.9. The value was gradually increased with increasing the ATH contents from 27.3 for 10 wt.% to 39.7 for 50 wt.% ATH, exhibiting an improvement of the flame retardancy of about 66%. This considerable increase in the LOI value indicated that ATH played an effective role in retarding the flammability of kenaf/PLA green composites. In fact, when the composite specimens containing ATH were flamed during the test according to ISO 4589 standard, it was seen that some water and water vapor were released from the testing specimen, and then, the released water was quickly boiled off. This phenomenon reflected that ATH acted as heat absorber and flame-retardant of the green composite, resulting in the increased LOI value, as similarly found in other literatures.[6,8]



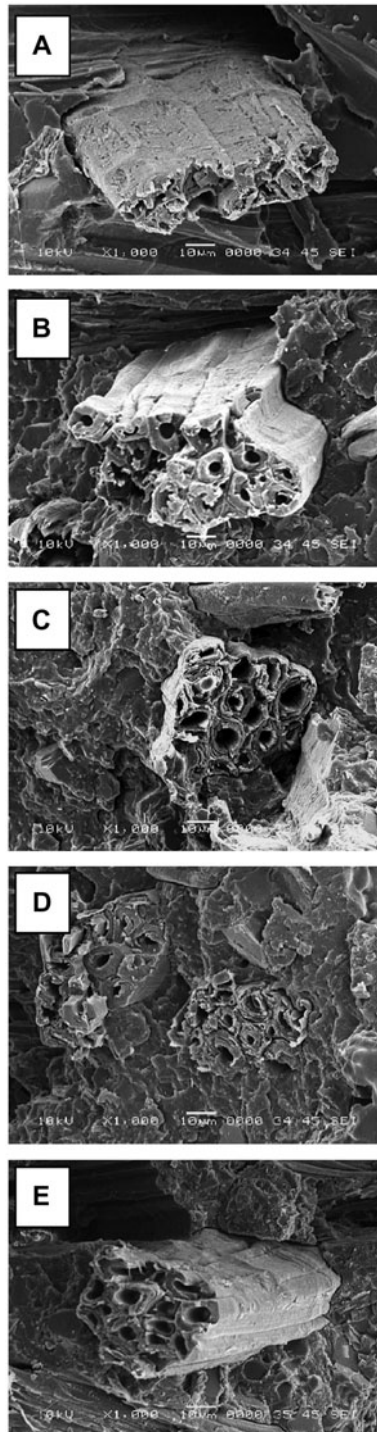


Figure 3. SEM images of the fractured surfaces of kenaf/PLA green composites without (A) and with various ATH loadings. (B) 10 kenaf/PLA, (C) 20 kenaf/PLA, (D) 30 kenaf/PLA, (E) 50 kenaf/PLA composites.

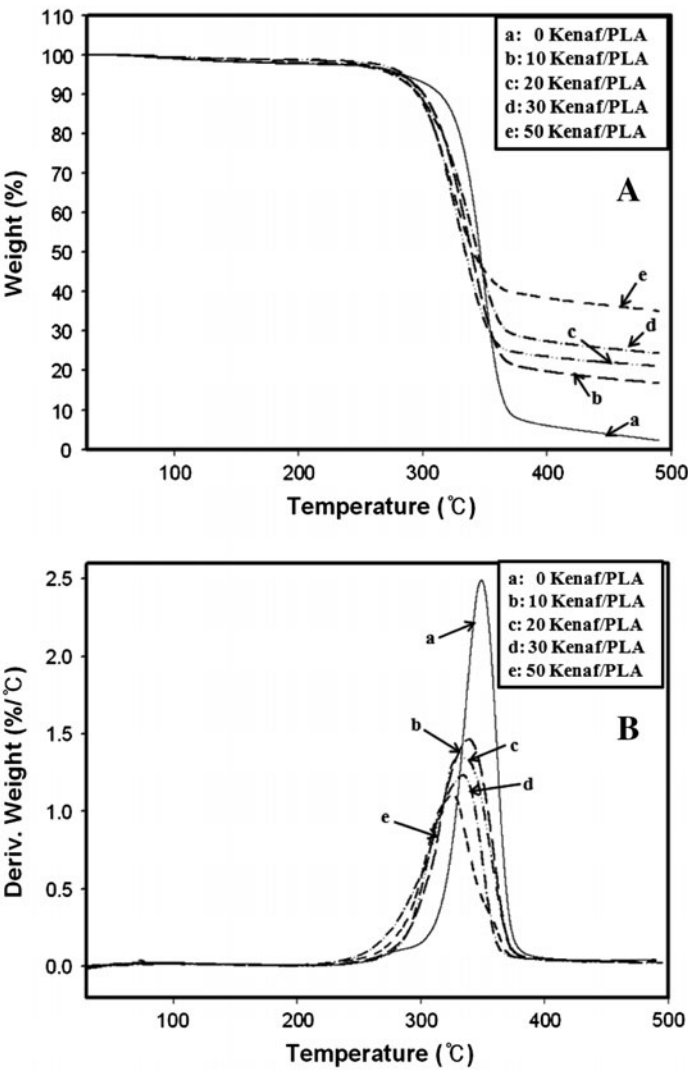


Figure 4. TGA (A) and DTG (B) curves measured in  $N_2$  for kenaf/PLA green composites without (a) and with various ATH loadings (b–e).

Table 2. A summary of compositions of PLA/ATH pellets and kenaf/PLA green composites.

Composites	PLA/ATH pellets		Kenaf/PLA green composites		
	PLA (wt.%)	ATH (wt.%)	Kenaf (wt.%)	PLA (wt.%)	ATH (wt.%)
0 Kenaf/PLA	100	0	40	60	0
10 Kenaf/PLA	90	10	40	54	6
20 Kenaf/PLA	80	20	40	48	12
30 Kenaf/PLA	70	30	40	42	18
50 Kenaf/PLA	50	50	40	30	30

Table 3. LOI values of kenaf/PLA green composites without and with various ATH loadings. The ATH contents were based on the PLA amount in the composite.

Green composites	LOI
0 Kenaf/PLA	23.9
10 Kenaf/PLA	27.3
20 Kenaf/PLA	30.0
30 Kenaf/PLA	31.3
50 Kenaf/PLA	39.7

Figure 5 displays photos of the specimens taken with a digital camera during LOI test of various kenaf/PLA green composites. The numbers designated in each specimen in the figures indicate the oxygen concentration (vol. %) used for each measurement of LOI test. The values 23.8, 27.2, 30.2, 31.4, and 39.6 in the fifth specimen of each figure, which correspond with the  $C_f$  described in the experimental section, indicate the final oxygen concentrations used for the samples A–E, respectively. The following describes the testing procedure. Given for the kenaf/PLA green composite specimens shown in Figure 5(A), the first specimen was ignited in the presence of 23.4% oxygen concentration in air and checked out the specimen loss of 5 cm for 3 min. Once the specimen was not consumed for the duration of 3 min, the first test was temporarily stopped, and then, the oxygen concentration was increased to 23.6% for the second specimen. This procedure was repeated with the increment of oxygen concentration by 0.2% until the 5 cm in length was consumed or lost from the top of each specimen for 3 min. The five specimens in each figure from A to E represent a serial of each composite specimen from the beginning to the end of LOI test, displaying their appearances at each testing stage. The far left specimen represents the beginning stage of the test and the far right one shows the ending stage of the test. The tested specimens in the corresponding testing stage looked similar, as seen in each figure from A to E. However, the amount of oxygen required for the LOI test was varied depending on flame-retarding capability of the composite specimen, resulting in different LOI values, as shown in Table 3.

### 3.4. Dynamic mechanical properties

Figure 6 shows the dynamic mechanical properties of kenaf/PLA green composites without and with ATH at various loadings. The storage modulus of the green composite was considerably increased with increasing ATH content, resulting in an improvement of about 136% (from 2.17 GPa for 0% ATH to 5.14 GPa for 50% ATH) of the modulus measured at room temperature, compared with the kenaf/PLA composite without ATH. It turned out that the incorporation of ATH into the PLA matrix made the composite stiffer. In other words, the ATH particles in the matrix restricted the mobility of PLA molecules. It can also be said that ATH played a critical role not only as flame-retardant but also as reinforcement. Of course, kenaf fibers contributed to increasing the dynamic mechanical property as reinforcement, leading to the PLA molecules less flexible.

The storage modulus was decreased at above 60 °C. Approaching the glass transition temperature near 66 °C (based on the  $\tan \delta$  result), the storage modulus was rapidly decreased down to about 90 °C. In the case of kenaf/PLA green composite without ATH, it was found that above about 90 °C the storage modulus was increased again due to recrystallization of PLA in the composite.[8] However, such the behavior was reduced in the presence of ATH in the composite. Interestingly, with increasing

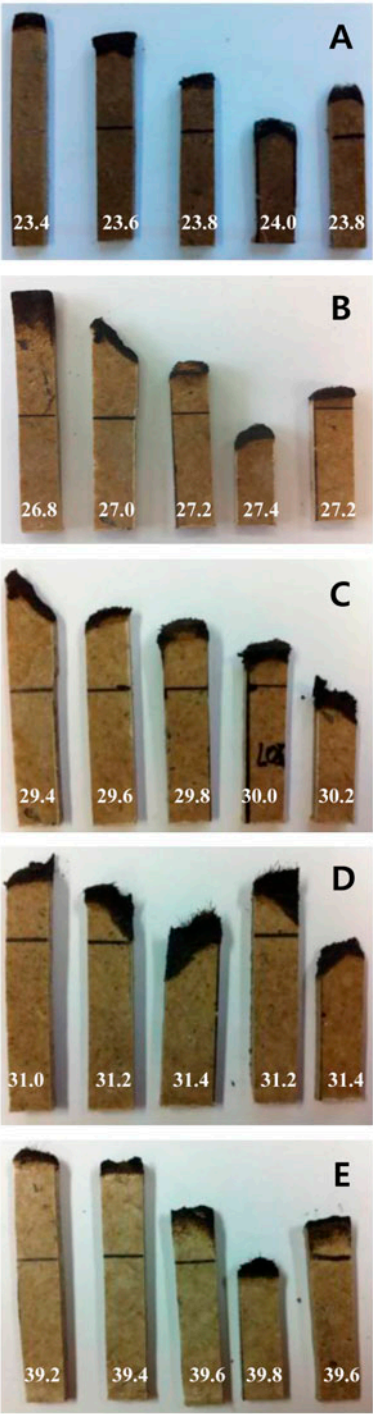


Figure 5. Digital camera photos of the specimens after LOI tests performed with kenaf/PLA green composites without (A) and with various ATH loadings (A–E).

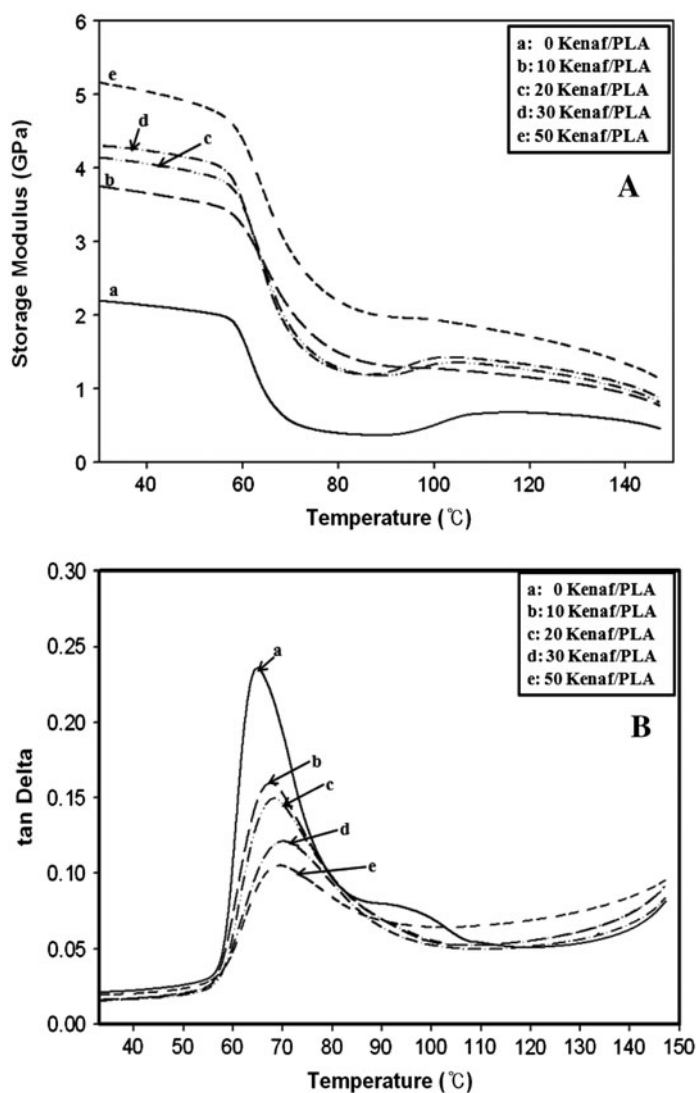


Figure 6. Variations of the storage modulus (A) and  $\tan \delta$  (B) of kenaf/PLA green composites without (a) and with various ATH loadings (b–e).

the ATH content, the recrystallization phenomenon was diminished, and then, it almost disappeared at 50 wt.% of ATH. The  $\tan \delta$  peak height, which is related with the damping characteristic of a polymer or composite material, was significantly decreased with increasing the ATH content, indicating a reinforcing effect of ATH. The  $\tan \delta$  peak temperature, which may be referred to as glass transition temperature, was slightly shifted to high temperature with increasing ATH contents in the PLA matrix. Therefore, it was concluded that an incorporation of ATH into the PLA gave rise to a combined effect of stiffening kenaf/PLA green composite and alleviating recrystallization phenomenon of PLA matrix in the composite, in addition to a flame-retarding effect.

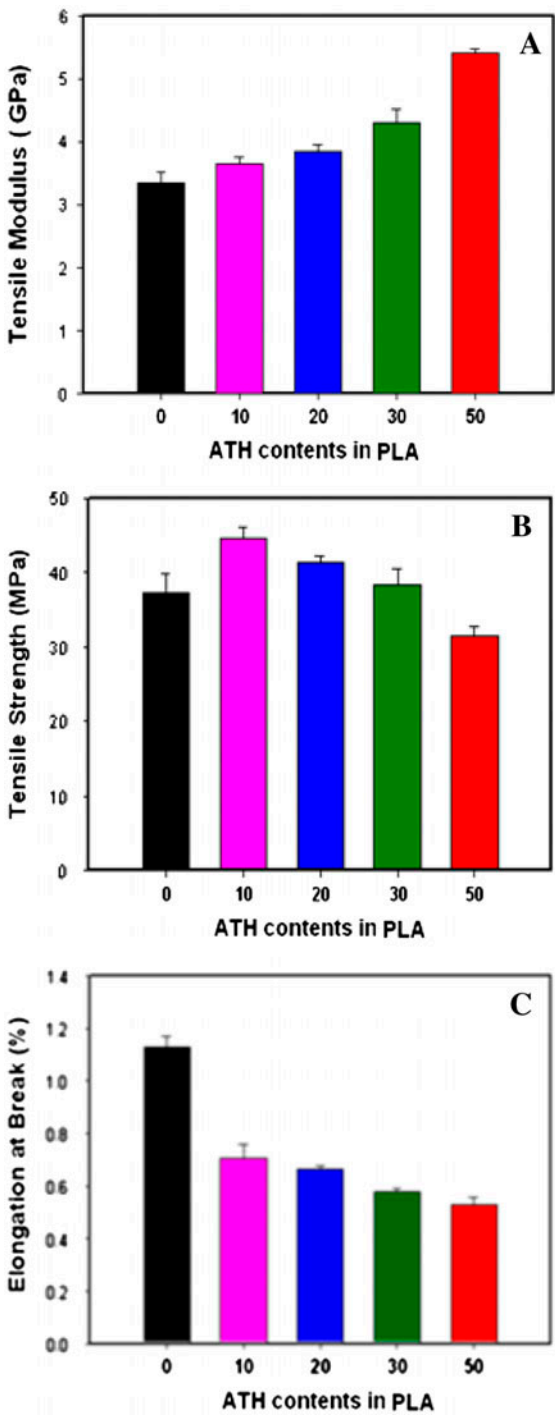


Figure 7. Comparisons of tensile modulus (A), strength (B), and elongation at break (C) of kenaf/PLA green composites with various ATH loadings.

### 3.5. Tensile properties

Figure 7 compares the tensile modulus and strength of kenaf/PLA green composites as a function of ATH contents in the PLA matrix. The tensile modulus was gradually increased with increasing the ATH content, indicating about 59% improvement from 3.4 GPa at 0 wt.% to 5.4 GPa at 50 wt.%. Such a considerable improvement was in good agreement with the dynamic storage modulus result above-mentioned. The elongation at break was gradually decreasing with increasing the ATH as well. At 50 wt.% ATH, the elongation was decreased by 50%, compared to the composite without ATH. The tensile strength of the composite without the ATH was increased about 16% at a 10 wt.% ATH loading in the PLA and then gradually decreased with increasing the ATH content to 50 wt.%. The decreasing tendency of the strength may be ascribed to ATH agglomeration in the PLA matrix in the composite, causing some microstructural defects, which are, in general, critical in determining the mechanical strength of a material. Such the agglomeration was not observed in the PLA/ATH specimens without using kenaf fibers. It was noted that the PLA contents in the PLA/ATH specimens were constantly 60 wt.%, whereas those in the composites were in the range of 30–54 wt.%, as listed in Table 2. Therefore, it was pointed out that an incorporation of ATH at 50 wt.% or higher into the PLA played a positive role in the modulus of kenaf/PLA green composites but a negative role in the strength, compared with the mechanical properties of kenaf/PLA composite without ATH.

## 4. Conclusions

Kenaf/PLA green composites with a flame-retardant ATH were successfully fabricated by means of a compression molding method using PLA/ATH pellets with various ATH loadings, which were processed using twin-screw extrusion technique. The flame retardancy of the green composite was significantly increased with increasing the ATH content, indicating an increase in LOI values. The improvement of the flame retardancy was about 66% compared with the pristine kenaf/PLA counterpart. It was noticeable that ATH played an effective role in retarding the flammability of kenaf/PLA green composites. The storage modulus was considerably increased with increasing the ATH content, whereas the  $\tan \delta$  value was decreased as well. The tensile modulus was markedly increased up to 50 wt.% ATH, whereas the strength was increased to 10 wt.% ATH and then decreased gradually to 50 wt.% ATH. It was concluded that an incorporation of ATH into PLA by extrusion technique considerably contributed to improving the dynamic storage modulus and tensile modulus as well as the flame retardancy of kenaf/PLA green composites, being supported by their fracture surfaces observed.

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