

# Effects of Areca Husk Fiber Loading and Alkaline Treatment on Properties of Natural Rubber Latex Foam

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## Abstract

The impact of integrating areca husk fiber (AHF) and employing alkaline treatment on the mechanical, thermal, and morphological characteristics of natural rubber latex foam (NRLF) is examined in this research. Areca husk fibers, derived from agro-industrial waste, were included in the NRLF matrix at different loadings (0, 5, 10, 15, 20, and 25 pphr). The fibers were chemically treated with sodium hydroxide (NaOH) to enhance surface roughness and augment interfacial interaction with the NRLF. Tensile tests indicated enhanced tensile strength at the appropriate filler load of 15 pphr for untreated AHF and 10 pphr for treated AHF. The elongation at break was decreased as fiber loadings increased, with greater elongation exhibited by treated AHF-filled NRLF compared to untreated AHF-filled NRLF. The foam's density and compression set increased with elevated fiber loadings; however, treated AHF-filled NRLF demonstrated improved elasticity and thermal stability compared to untreated samples. Filler-matrix interactions were validated through scanning electron microscopy (SEM), which showed reduced cell sizes and more homogeneous foam formations in the treated AHF samples. The results indicate that the efficacy of NRLF is enhanced by the alkaline treatment of AHF, rendering it an appropriate material for sustainable composite applications.

**Keywords:** Areca husk fiber, Natural rubber latex foam, Natural rubber, Alkaline treatment, Natural fiber

## Introduction

In the past few years, environmental contemplations and public consideration have become progressively significant as the world endeavours toward ecological quality and conservation through sustainable development and cleaner technology. Natural fibers (NF) are particularly important as reinforcement in environmentally friendly polymer composite preparations, as these NFs have great benefits such as strength and stiffness. It has good performance while being low-cost, renewable, and biodegradable. As a result, mixing polymers with NFs like sisal or jute can result in entirely green blends consisting of renewable materials [1]. NFs have high specific strength, modulus, and failed elongation, which make them a viable material substitute for conventional fillers like silica,

glass, and calcium carbonate [2]. NFs have high specific strength, modulus, and failed elongation, which makes them a viable material substitute for conventional fillers like silica, glass, and calcium carbonate. Widely used, such inorganic fillers are also expensive when compared to natural additives. Thus, NFs are progressively utilized as a filler in rubber [3], plastics [4-6], epoxy resin [7-9], and thermoplastic elastomers [10]. Organic waste materials such as grain straw and cotton waste [11], kenaf [12], wood fiber [13], bamboo waste [14,15], areca husk waste [16-18], oil palm waste [19], and sugarcane bagasse waste [20] were widely used for manufacturing NFs composite materials.

Natural rubber latex (NRL) is the primary latex used in industrial applications. NRL is defined as the

dispersion of NRL particles in water. The pH level and the surrounding media's ionic conditions influence the colloid's stability in latex. NRLF is a cellular rubber derived from NRLF. The Dunlop process is specifically designed to efficiently manufacture thick-molded NRLF components, including pads, cushion pads, sleepers, and upholstery.

AHF is an NF that is not expensive, abundantly available, and can be found in agro-industry waste [17]. AHF, extracted from the areca fruits, is presently used as housing insulation material and value-added products such as cushions, handcraft, and nonwoven fabrics [21]. Combining NFs and polymers leads to engineered green composite materials [22-25]. Recently, the utilization of NFs in NRLF has attracted many researchers' attention because there are great opportunities for its use in various applications and being an environmentally friendly green material. In recent years, researchers have discovered that organic waste and NFs such as oil palm fiber [26,27], kenaf [28-30], rice husk powder [31], bagasse [27], jute fiber [32], bamboo leaf fiber [33] and eggshells [34] have been used successfully as fillers in NRLF. In addition, some works on AHF suggest that the compatibility between AHF and the matrix (natural rubber), structure, and surface of AHF can be improved by chemical or physical treatment of AHF [35] in the dry rubber compound. However, to our knowledge, no study has reported the use of AHF in NRLF.

Hence, AHF was chosen in this study to investigate the effect of AHF loading and the alkaline treatment on the mechanical, thermal, and morphological properties of AHF-filled NRLF foam properties. The study focuses on assessing how different AHF loadings impact the tensile strength, elongation, density, and compression set of NRLF, with particular attention to the improvements brought by alkaline treatment. The research also explores the role of alkaline treatment in enhancing the fiber's surface roughness and interfacial bonding with the NRLF matrix, contributing to improved mechanical strength and foam structure.

Additionally, the study examines the thermal stability and degradation resistance of treated versus untreated AHF-filled NRLF, hypothesizing that treated AHF will provide better thermal and structural performance due to finer, more uniform foam cells.

### Materials and methods

The components and chemicals required to prepare NRLF are presented in **Table 1**. High ammonia natural rubber latex (HANRL) with a dry rubber content of around 60 wt%, produced by T.T. Latex & Products Co., Ltd. in Nakhon Si Thammarat, Thailand, served as the raw material for the manufacturing of NRLF. Sulfur, Wingstay L, potassium oleate, zinc diethyldithiocarbamate (ZDEC), zinc 2-mercaptobenzthiozolate (ZMBT), diphenylguanidine (DPG) and sodium silicofluoride (SSF) were purchased from Bayer Thai Co., Ltd. Sodium hydroxide (NaOH) was supplied by RCI labscan Ltd., Thailand. NaOH was used to increase the surface roughness of the AHF and improve the interfacial bonding between the NRLF and AHF.

The areca fruits or betel nuts are available everywhere in Nakhon Si Thammarat Province, South of Thailand. For this research, areca fruits were collected from a local of Phra Phrom district, Nakhon Si Thammarat Province. Areca fruits were separated from husk by a manual cutter. AHFs were cut into an average size of 5 mm. The husks of Areca fruit were soaked and immersed in water at room temperature for 48 h to loosen the fibers from the hard shell of the husk. AHFs were then immersed in 15 % NaOH solution for 12 h at ambient temperature. Then, the AHFs were washed and immersed in distilled water to remove residual NaOH from the AHFs. The washing process was repeated until the pH of the water was 7. Then, AHFs were dried at 60 °C for 48 h. Surface textures of AHF before and after treatment were investigated with a Scanning Electron Microscope (SEM, ZEISS, MERLIN COMPACT) at 500× and 700×.

**Table 1** Compounding formulation of untreated and treated AHF-filled NRLF.

Ingredients	Total Solid Content (%)	Formulation (pphr)
Natural rubber latex	60	100
K-Oleate	20	1.0
Sulfur	50	5.0

Ingredients	Total Solid Content (%)	Formulation (pphr)
ZDEC	50	1.0
ZMBT	50	1.0
Wingstay L	50	1.0
ZnO	50	5.0
DPG	33	1.0
SSF	20	1.5
Untreated AHF, treated AHF	100	0, 5, 10, 15, 20, 25

### Foam sample preparation

Initially, HANRL was subjected to filtration and weighed following the formulation. Subsequently, HANRL and the compounding ingredients (ZMBT, ZDEC, Sulphur, Wingstay L, and potassium oleate) were stirred for approximately 4 h using a mechanical stirrer at 65 rpm. Following 4 h of uninterrupted stirring, the HANRL compound was aerated utilizing the OTTO Mixer HM-273. The untreated AHF and treated AHF were gradually included in the HANRL compound, and the combination was blended with a stand mixer at 75 rpm for approximately 4 min until a homogeneous compound was achieved. Subsequently, the HANRL compound was subjected to intensive beating for approximately 2 min at 100 rpm until its volume expanded to 3 times the starting volume. The foaming speed was reduced to 75 rpm after reaching the necessary volume to achieve a fine and uniform foam. Subsequently, continuous speed at 75 rpm the principal gelling agent (zinc oxide, ZnO) was incorporated with diphenyl guanidine (DPG) into the foam, and the mixing persisted for an additional minute. Subsequently, the secondary gelling agent (sodium silicofluoride, SSF) was incorporated, and the foam was agitated for an additional minute to achieve a uniform, ungelled foam. The ungelled foam was immediately poured into 2 aluminium moulds, 250×200×2 mm<sup>3</sup> and 50×50×25 mm<sup>3</sup>. Three samples were prepared for each size and allowed to gel for 3 min at room temperature. The gelled foam was subsequently cured in a steam environment at 100 °C for 30 min. The AHF-filled natural rubber latex foam (ANRLF) was removed from the mould and thoroughly rinsed with distilled water to eliminate excess unreacted chemicals. After washing, the cured ANRLF was dried in a hot air oven at 60 °C for 24 h. The thoroughly dried foam will exhibit an off-white hue.

### Measurement of tensile properties

Tensile tests followed ASTM D 412 with a Hounsfield-H10 KS universal testing machine. Dumbbell-shaped specimens were extracted from the ANRLF sample with a Pneumatic Shape Cutting Device 403S-S Series. Five samples were analyzed from each batch of AHF. Tensile testing was conducted at room temperature with a 500 mm/min crosshead speed. Both untreated and treated samples of AHF-filled NRLF with filler loadings of 0, 5, 10, 15, 20, and 25 pphr were evaluated. Tensile strength and elongation at break were determined through testing, and the average of 5 tests was reported.

### Foam density

The density of untreated AHF and treated AHF-filled NRLF was calculated by dividing mass by specimen volume, as demonstrated in Eq. (1). The samples utilized in this testing procedure were of a regular shape with a minimum volume of 1000 mm<sup>3</sup>, following ASTM D3574. Five samples for each AHF loading were analyzed, and an average of 3 outcomes was documented.

$$\text{Density} \left( \frac{\text{kg}}{\text{m}^3} \right) = \left( \frac{M}{V} \right) \times 10^6 \quad (1)$$

where  $M$  is the mass of the specimen (g), and  $V$  is the volume of the specimen (mm<sup>3</sup>).

### Compression set

The compression set characteristics of untreated AHF and treated AHF-filled NRLF were assessed following ASTM D3574. The samples utilized were of standard dimensions, measuring 50×50×25 mm<sup>3</sup>, including parallel top and bottom surfaces and sides that are practically perpendicular. Five specimens were

examined for each sample. Specimens were positioned in the testing device and deflected to  $50 \pm 1$  % of their original thickness. After 15 min, the deflected specimens and apparatus were positioned in a mechanically convected air oven for 22 h at a test temperature of  $70 \pm 2$  °C. Specimens were promptly extracted from the device and measured after a 30-minute recovery period. Three samples were evaluated for each AHF loading. The compression set is shown as a percentage of the initial thickness, as seen in Eq. (2).

$$C_t = [(t_o - t_f)/t_o] \times 100 \quad (2)$$

where  $C_t$  is the compression set expressed as a percentage of the original thickness,  $t_o$  is the original thickness of the test specimen, and  $t_f$  is the final thickness of the test specimen.

### Scanning electron microscopy

The surface morphology of untreated AHF, treated AHF, and both untreated and treated AHF filled with NRLF was examined using a scanning electron microscope (ZEISS, MERLIN COMPACT). The fibers and foam surfaces were affixed to aluminium stubs and sputter-coated with a small layer of gold to mitigate electrostatic charge and enhance image resolution during analysis. The morphology of untreated AHF, treated AHF, and both untreated and treated AHF-filled NRLF was examined.

### Thermal analyses

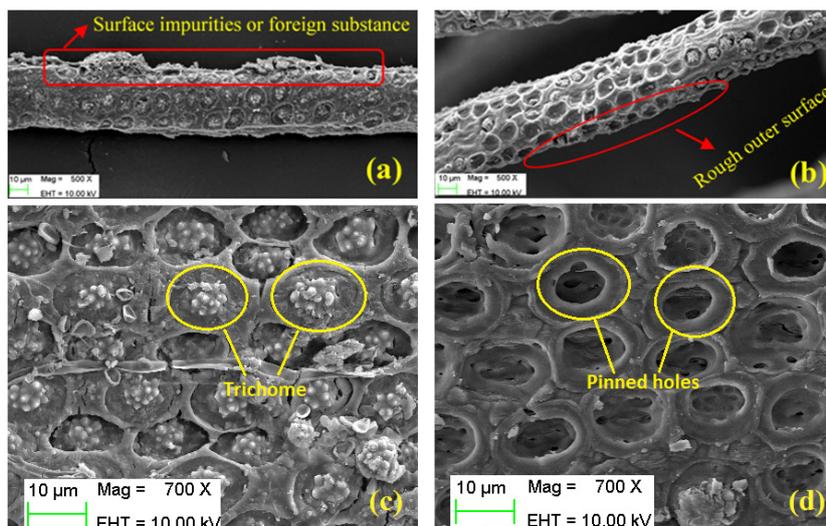
Thermal examination of untreated AHF and treated AHF-filled NRLF samples was conducted using a Thermogravimetric Analyzer (TGA4000, PerkinElmer). TGA analysis was performed in ramp mode at a heating rate of 10 °C/min, ranging from 30 to 800 °C under nitrogen atmosphere. The original sample weight was around 4 mg. The TGA thermograms were examined for their initial decomposition temperatures.

## Results and discussion

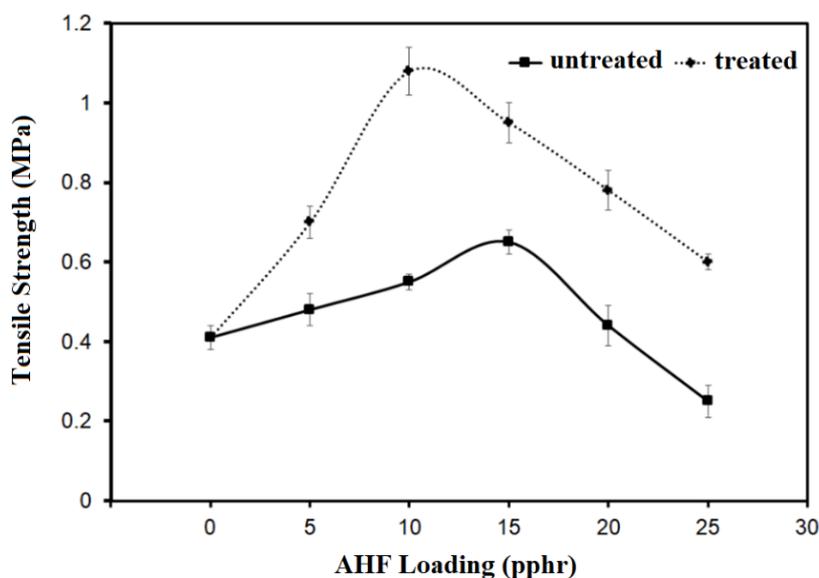
### Surface treatment

Hydrophobic materials (matrix) and hydrophilic (dispersed) fibers are often difficult to get along. As a result, the ability of the composite to transfer stress between the 2 phases decreases. The process of alkaline fiber surface treatment stimulates the surface of the fiber to have roughness at the fiber/matrix interface. The alkaline treatment changes the structure's hydrogen bonds and gets rid of some of the lignin, waxes, and oils to show short crystals. Fibers that have been treated with alkaline skin will have an increased concentration of cellulose at the touch and skin roughness, resulting in better matrix coordination [36]. Alkaline treatment of AHF fiber surfaces leads to the development of the Fiber-cell-O-Na group within the molecular chains of cellulose. The hydrogen bonds within the cellulose structure are removed. The interactions between cellulose molecular chains form new hydrogen bonds, reducing the density of the cellulose crystal structure. The reduced hydrophilic hydroxyl groups increase the moisture resistance and increase the surface area of the fiber. Chemicals can penetrate the cellulose molecules more easily. This results in better compatibility between the fiber and the matrix [37].

The SEM image of NaOH-treated AHF is shown in **Figures 1(b)** and **1(d)**, and it clearly shows a large number of pinholes on the surface due to the elimination of fatty coats from the fiber. NaOH treatment of areca fiber eliminates waxy tissue, adhesive pectin, and hemicelluloses [38]. A comparison between the untreated and NaOH-treated AHF exhibits topographical changes. The elimination of lower molecular weight organic compounds in the AHF results in a rough outer surface formation. This reduces moisture absorption and provides an effective surface area for good adhesion to the matrix. The alkaline treatment of AHF showed a rougher surface than that of untreated AHF (**Figure 1(c)**). The observation of alkalinity conditions which dissolved the lignin naturally present in AHF, leaving a hollow structure on the surface (**Figure 1(d)**), was also found in other studies [27].



**Figure 1** SEM micrographs of the AHF surfaces before and after NaOH treatments: (a) untreated and (b) treated AHF at 500× magnification, (c) untreated and (d) treated AHF at 700× magnification.

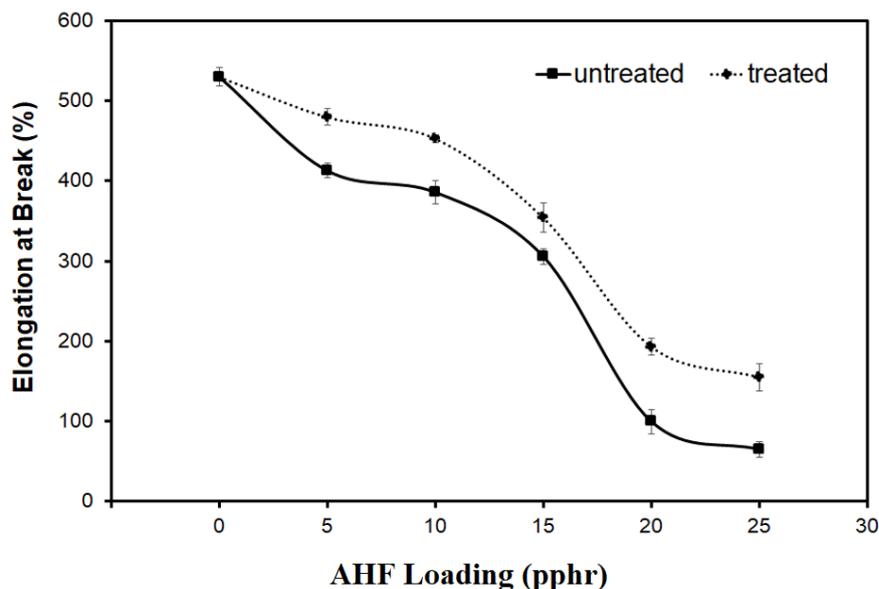


**Figure 2** Tensile strengths of untreated and treated AHF-filled NRLF at different AHF loadings.

### Tensile properties

**Figure 2** shows the tensile strength of untreated and treated AHF-filled NRLF at different levels of filler loadings. For both untreated and treated AHF-filled NRLF conditions, the tensile strengths were improved up to 15 pphr (untreated) and 10 pphr (treated) filler loadings. However, the strength decreased continuously as the loading increased higher than 15 pphr (untreated) and 10 pphr (treated) up to 25 pphr filler loadings. The tensile strength of samples with untreated AHF increased from 0.41 MPa (control) to 0.65 MPa (15 pphr) and then decreased to 0.25 MPa (25 pphr). On the

other hand, by using treated AHF, the tensile strength increased from 0.41 MPa (control) to 1.08 MPa (10 pphr) and then decreased to 0.6 MPa (25 pphr). As shown in **Figure 1**, AHF-filled NRLF with treated AHF have a higher tensile strength at lower filler loading than samples with untreated AHF. The increase in efficiency was due to the reduction in the cell size of the foam, which can be observed in the following SEM results (**Figure 6**). Alkaline treatment on AHF increased the roughness of fiber surfaces. The increase in the surface roughness results in better mechanical coordination between AHF and the latex matrix of the foam [28,39].



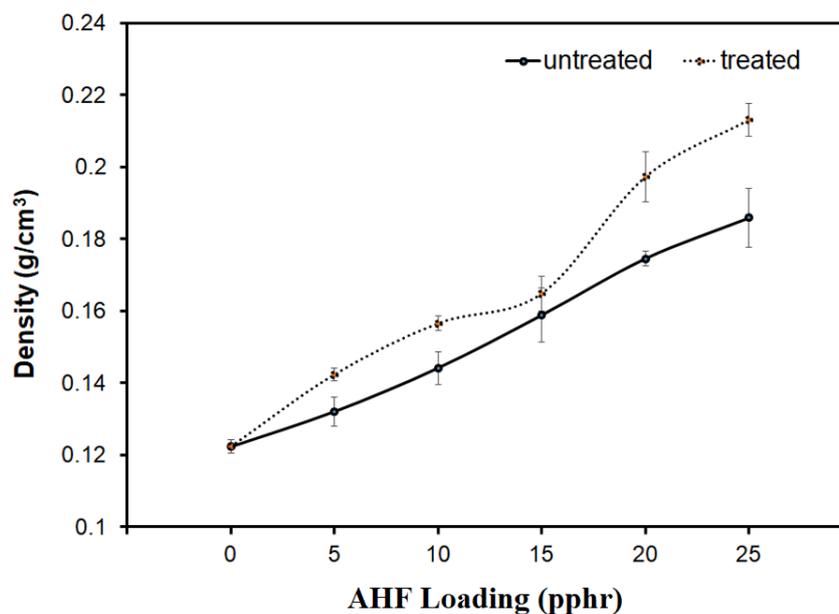
**Figure 3** Elongation at break of untreated and treated AHF-filled NRLF at different AHF loadings.

**Figure 3** shows the elongation at break for untreated and treated AHF-filled NRLF at different filler loadings. Higher filler loadings decrease the elongation at break for both conditions due to the rise of stiffness and brittleness of NRLF in the presence of filler [40]. For treated AHF-filled NRLF, the elongation at breaks was higher than the elongation at break for untreated AHF-filled NRLF. The elongation at break for untreated AHF-filled NRLF reduced from 530 % (0 pphr AHF) to 65 % (25 pphr AHF), while treated AHF-filled NRLF reduced from 530 % (0 pphr) to 155 % (25 pphr). According to prior study conducted in 2014, alkaline-treated AHF demonstrated enhanced elongation at break compared to untreated AHF fibers. The enhancement in elongation at break of alkaline-treated AHF may possibly be attributed to alterations in cellulose structure resulting from the alkaline treatment [41]. As a result, the treatment improves the elongation at break of the NRLF.

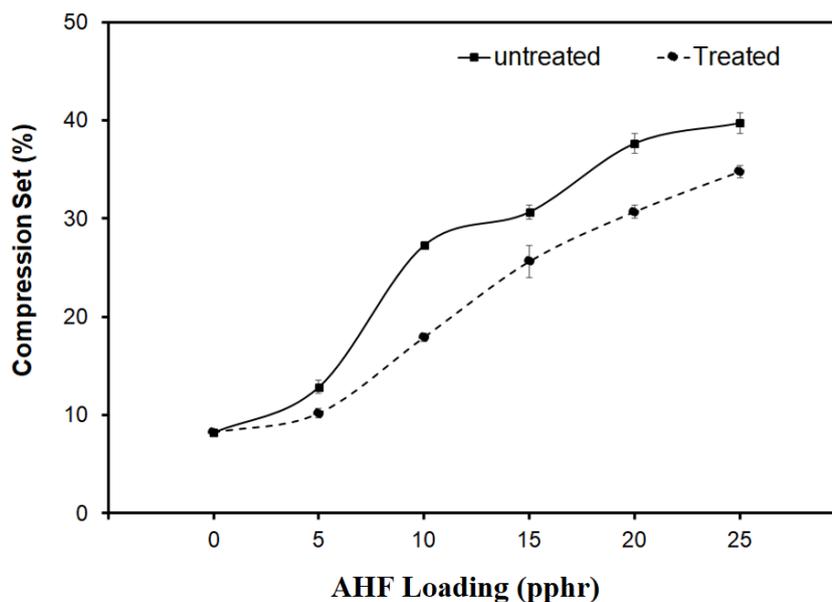
#### Foam density

**Figure 4** shows the density for untreated and treated AHF-filled NRLF. The density for both conditions increased with an increase in AHF loadings. The density of untreated AHF-filled NRLF increased

from 0.1223 to 0.1859 g/cm<sup>3</sup>, while the density of treated AHF-filled NRLF increased from 0.1223 to 0.2132 g/cm<sup>3</sup> with the maximum addition of fillers, 25 pphr. The filler increases the mass of NRLF, which leads to an increase in the density of AHF as the loading increases. However, treated AHF-filled NRLF have a higher density than untreated AHF-filled NRLF due to the cell size factor and the alteration on the fiber surfaces. The cell size of the treated AHF-filled NRLFs was smaller and more uniform than the untreated AHF-filled NRLFs, as shown in **Figure 6**. The decrease in cell size creates a more compact and higher-density foam. As discussed before, the decrease in cell size was due to the better filler matrix interaction between AHF and latex. To reduce the defect point during the formation of NRLF, the filler matrix interaction is important in producing a very uniform cell size of NRLF [28]. The combination of NaOH-treated AHF and the NRLF matrix increases the composite's density. The enhancement results from stronger fiber-matrix adhesion and a more compact structure due to the alkaline treatment. This diminishes the volume of voids inside the material, resulting in a more homogeneous and dense composite [6].



**Figure 4** Density of untreated and treated AHF-filled NRLF at different AHF loadings.



**Figure 5** Compression set of untreated and treated AHF-filled NRLF at different AHF loadings.

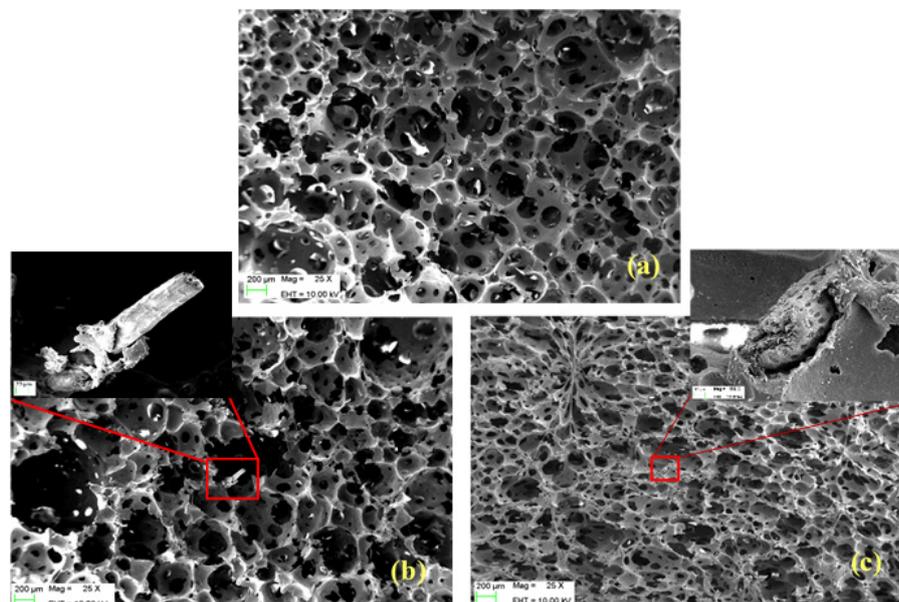
### Compression set

Compression set is a measurement of the elastic behavior of the material. **Figure 5** shows the compression set value for untreated and treated AHF-filled NRLF. The compression set value in **Figure 5** shows that the control NRLF has the lowest percentage, indicating that the control foam has the highest elasticity. The compression set increases as the AHF fiber loadings with both conditions increase. This was due to the NRLF losing its elasticity by having filler

agglomerates, thereby restricting the molecular chains' flexibility and increasing the stiffness [29]. From the results, it was found that the untreated AHF-filled NRLFs have higher compression set values compared to the treated AHF-filled NRLFs. This is likely due to the clean surface of treated AHF free from pectin, hemicellulose, and other impurities. The surface of treated AHF could interact with NRLF better, resulting in more homogenous filler incorporation than untreated AHF-filled NRLF. The linear increase of compression

set percentages as the loading increases for the treated AHF shows the well-distributed fillers compared to the untreated samples. As a result, the treated AHF-filled

NRLF were more elastic than the untreated AHF-filled NRLF.



**Figure 6** Surface micrograph (a) micrograph of NRLF (control), (b) micrograph of untreated AHF filled NRLF, and (c) micrograph of treated AHF filled NRLF.

### Morphology

The dispersion and distribution of untreated and treated AHF in NRLF were studied using SEM. The SEM micrographs of NRLF (control sample), untreated AHF-filled NRLF, and treated AHF-filled NRLF were shown in **Figures 6(a) - 6(c)**, respectively. In all the figures, the image clearly shows open-cell structures [30]. The treated AHF-filled NRLFs show smaller pores and stable cell structure sizes compared to untreated AHF-filled NRLFs. This confirmed that adding treated AHF to the system decreased cell size and provided

better cell structures. **Figures 6(b) - 6(c)** show the filler-matrix interaction of untreated and treated AHF-filled NRLF at 10 pphr AHF loading. From **Figure 6(b)**, the untreated AHF showed weak filler interaction with NRLF, while treated AHF-filled NRLF in **Figure 6(c)** shows good filler interaction with foam, leading to the improved properties of NRLF. This is an indication that the alkaline treatment was able to increase the interaction between AHF and NRLF interfaced. These results are consistent with previous studies [27,28].

**Table 2** Thermal stability parameters of untreated and treated AHF filled NRLF at 10 pphr loading.

Sample Code	Degradation Temperature (°C)		Maximum weight loss (%)	Temperature at maximum weight loss (°C)	Final char residue (%)
	T <sub>d5</sub>	T <sub>d10</sub>			
NRLF (Control)	297.00	338.85	98.94	379.43	1.06
Untreated AHF-filled NRLF	277.75	323.55	96.81	379.64	3.19
Treated AHF-filled NRLF	309.43	341.33	94.06	380.66	5.94

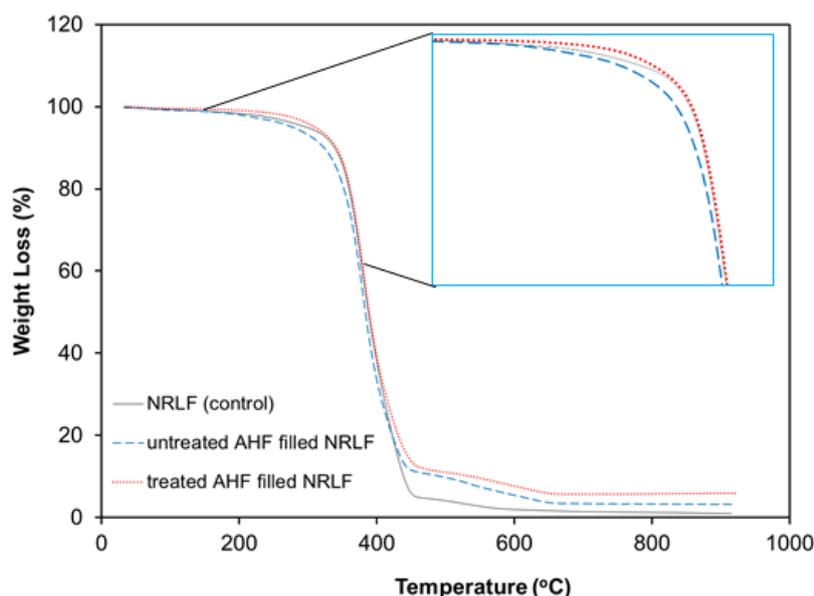
### Thermogravimetric analysis

Thermal stability is the ability of a material to maintain desired properties, such as strength, toughness,

or flexibility, when the temperature changes. Understanding the details of polymer degradation when heat is applied leads to designing materials with

improved properties for specific applications [42]. The results of the thermogravimetric analysis (TGA) of NRLF (control), untreated AHF-filled NRLF, and treated AHF-filled NRLF are shown in **Table 2** and **Figure 7**. The weight change of AHF-filled NRLFs with increasing temperature under nitrogen gas was measured, and a weight loss chart as a function of the temperature was considered. Treated AHF-filled NRLF (10 pphr) showed higher thermal stability compared with untreated AHF-filled NRLF (10 pphr) and NRLF

(control) due to the better interaction between AHF and NRL. **Table 2** shows the temperature at 5 and 10 % weight loss, the maximum weight loss, the temperature at the maximum weight loss, and the final char residue percentages. The temperature at 5 and 10 % weight loss of treated AHF-filled NRLF showed higher degradation temperature than NRLF (control) and untreated AHF-filled NRLF, respectively. The AHF-filled NRLF produced a higher char residue than the NRLF (control).



**Figure 7** TGA curved of untreated and treated AHF filled NRLF at 10 pphr loading.

## Conclusions

The study found that incorporating AHF into NRLF, especially with alkaline treatment, significantly enhanced its properties. Treated AHF improved tensile strength by 163.4 %, reaching 1.08 MPa at 10 pphr, while untreated AHF showed a 58.5 % increase at 15 pphr. Treated AHF also retained better flexibility, with elongation at break decreasing to 155 % compared to 65 % for untreated fibers at 25 pphr. Foam density increased by 74 % for treated AHF and 52 % for untreated AHF. Treated AHF also exhibited better elasticity with a lower compression set and improved thermal stability, with a degradation temperature 11.4 % higher than untreated samples. These improvements suggest that treated AHF enhances the mechanical, thermal, and structural properties of NRLF more effectively than untreated AHF. The SEM study of the

NRLF revealed an open cell structure with small pores and a uniform structure following AHF treatment. The untreated AHF-filled NRLF showed weak filler interactions with NRLF, while the treated AHF-filled NRLF showed good filler interactions with foam. Overall, the NRLF with treated AHF has better properties than those with untreated AHF. This research shows potential for sustainable applications, such as eco-friendly construction materials and automotive components, with enhanced mechanical and thermal properties. Future research should focus on optimizing fiber treatments, exploring additional natural fibers, and evaluating long-term durability for broader use in green technologies.

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