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Effect of Alkaline Treatment on Physical and Tensile Properties of Areca Leaf Sheaths (ALSs) used for Biodegradable ALS Plate Production

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Highlights:

- An interesting way of reducing molding due to moisture in ALSs in ALS plate production is alkaline treatment.
- Alkaline treatment with NaOH and $NaHCO_3$ increased the moisture resistance properties of ALSs.
- ALS treated with 1% NaOH and 1% $NaHCO_3$ can be properly used for ALS plate production.

Abstract. This research evaluated the possibility of alkaline treatment to solve the problem of molding due to moisture during storage of areca leaf sheaths (ALSs) to be used for biodegradable ALS plate production. The effect of alkaline treatment on the properties of ALSs was studied. Sodium hydroxide (NaOH) and sodium bicarbonate ($NaHCO_3$) were used as chemicals for surface treatment. The solution of NaOH and $NaHCO_3$ was varied at 1%, 3%, and 5% w/v. Surface treatment with NaOH and $NaHCO_3$ partially removed hemicellulose, cellulose, lignin, and impurities from the surface of the ALSs and increased the moisture resistance property of the ALSs. Treatment with NaOH made the ALSs' surface rougher, whereas treatment with $NaHCO_3$ had no effect on the surface of the ALSs. The decomposition temperature (T_{d2}) of the NaOH-treated ALSs decreased by approximately 7.0 to 10.5%, while for the $NaHCO_3$-treated ALSs it changed only slightly. The overall tensile properties of the NaOH-treated ALSs were better than those of the $NaHCO_3$-treated ALSs. The surface treatment with 1% NaOH and 1% $NaHCO_3$ increased the tensile modulus by 20.5% and 6.2%, respectively, as compared with the nontreated ALS. It was found that surface treatment with 1% NaOH and 1% $NaHCO_3$ could create suitable conditions for ALS plate production. This work is a preliminary study; more research still needs to be done.

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Effect of Alkaline Treatments on Physical and Tensile Properties of Areca leaf sheaths (ALSs) used for Biodegradable ALS plate Production

Keywords: *alkaline treatment; areca leaf sheaths; biodegradable packaging; biodegradable plate; disposable plate.*

1 Introduction

Areca palm tree, or *Areca catechu*, is a species of palm which is available and grows widely across Asia, for example, in India, China, Malaysia, Taiwan, Thailand, etc. [1]. The areca leaf sheaths (ALSs) are the portion of the plant that supports the connection between the branches and the trunk, as shown in Figure 1 [2]. Poddar, *et al.* [3] reported that the main chemical composition of ALSs consists of α-cellulose (66.08%), lignin (19.59%), and hemicellulose (7.40%). The minor constituents of ALSs are fatty oils and wax (5.06%), pectin (1.15%), and aqueous extract (0.72%). ALS fiber is composed of a small unit of cellulose surrounded and connected by hemicellulose and lignin [3]. Environmental problems can be reduced by using biomass-based polymers, which are ideal for many applications, such as product packaging [4-5]. ALS plates are also interesting for biomass packaging. Their usage in biodegradable, compostable, and ecofriendly food containers in India has been reported [6-9]. Nikhil, *et al.* [10] produced a disposable ALS plate by using a simple and energy efficient production process. The raw ALSs falling from the tree are collected and require a sun drying and storage for about six months. A cost-effective solar dryer has been developed to shorten the ALSs drying time [10]. In the ALS plate manufacturing process, the ALSs are first washed and brushed manually in water. The ALSs will gain some moisture from this step to make them more flexible during the pressing process. Then, the obtained ALSs are placed into a hot-pressing machine at a certain temperature and at sufficient time for its structure to be shaped according to the mold. The moisture content of the ALSs in this step is very important. It should be maintained at above 5% to prevent cracking of the plate [8].

Several applications of ALSs have been reported in Thailand. Jarawae [11] developed insulation made from ALSs. Junlacoupt & Srisuwan [12] developed a wickerwork bag from ALSs and studied its physical properties. The design and development of food packaging made from ALSs has been done by Sutthiyapiwat & Phayoonpun [13]. Three types of ALS food packages (in the shape of a square plate, a round plate, and a cup) were prepared. The only brand well-known for manufacturing ALS plates in Thailand is VEERASA from Nakhon Ratchasrima province. However, their productivity is still insufficient to meet the demand. Therefore, many small enterprises which grow areca trees in the area are interested in setting up ALS plate production facilities. The community in Nong Bua Sub-district, Ratsada, Trang, South Thailand, is interested in doing this also. However, they still have a problem with molding related to the humidity of the

ALSs in the storage step, because the south of Thailand is an area with a hot and humid climate. A method to reduce the humidity of the ALSs during storage is necessary.

Alkaline treatment has been used to reduce water absorption [14-15] and improve adhesion between natural fibers and polymers [16]. Sodium carbonate treatment can improve the adhesion properties between the ALS fiber and the bio polymer surface, and it also reduces water absorption [14]. Sodium hydroxide treatment of areca empty fruit fiber reduces hydrophilic hydroxyl groups in the fiber and increases the fiber's moisture resistance properties [15]. The effect of NaOH treatment of ALSs on the properties of epoxy composite reinforced ALSs processed by manual lay-up followed by compression molding was studied by Ashok, *et al.* [17]. They found that the surface modification of the ALSs increased the mechanical properties of the NaOH-treated ALSs reinforced composites [17]. Therefore, alkaline treatment with sodium hydroxide (NaOH) and sodium bicarbonate ($NaHCO_3$) to improve moisture resistance properties of ALSs in the storage step of disposable ALS plate production is interesting to solve the molding problem.

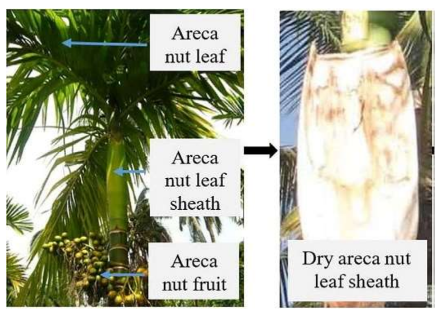

Figure 1 The position of areca leaf sheath (ALS) on the areca palm tree and dry ALS [2].

Some studies on ALS disposable plate processing have reported the chemical composition [3], physical properties [9,18], mechanical properties [9,19] and microstructure of ALSs [8]. Factors of ALS plate processing, such as the moisture content of ALSs and the temperature during the process, have been studied by Kalita, *et al.* [20]. Meanwhile, Jong, *et al.* [8] studied the limitations of the workable surface of ALSs due to the concavity in the middle and folds in raw ALSs. This was solved by applying a flattening process before the forming process. The tensile properties of the ALSs before and after flattening were also studied [8]. To our knowledge, no previous study has been done to increase the moisture resistance property of raw ALSs used in ALS plate production.

The objective of this work was to study the effect of type and chemical concentration in the alkaline treatment of ALSs. Different solutions of NaOH and $NaHCO_3$ were used. The physical properties, including dimension, thermal properties and morphological properties, and the tensile properties of nontreated and treated ALSs were determined.

2 Materials and Methods

2.1 Materials

The areca leaf sheaths (ALSs) used in the present study were obtained from Nong Bua Sub-district, Ratsada, Trang, Thailand, where the community is planning to start a compostable ALS plate production facility. Sodium hydroxide (NaOH) and sodium bicarbonate ($NaHCO_3$) were purchased from Sigma-Aldrich Co., Ltd., Bangkok, Thailand.

2.2 Surface Treatment of ALSs

NaOH and $NaHCO_3$ solutions were prepared at concentrations of 1%, 3% and 5% w/v. Each ALS was cut into 20 cm of length at the original width. Then, the ALS sample was soaked in the alkaline solution for 1 hr at room temperature, after which the treated ALS was washed with tap water until pH was neutral. It was then dried in the atmosphere under the sun until a constant weight was obtained. After treatment with NaOH or $NaHCO_3$, the surface of the ALS was characterized using Fourier transform infrared spectroscopy (FTIR). FTIR spectra were recorded with a Bruker ALPHA spectrometer in attenuated total reflection mode. The scanning range was set from 4,000 to 400 cm^{-1} with a scanning resolution of 4 cm^{-1} and 64 scans per sample. The obtained treated ALSs were used for physical, thermal, and tensile testing.

2.3 Physical and Thermal Properties Testing of the ALSs

The average width and length of the ALSs were measured by tape measure and the thickness of the ALSs was measured by digital vernier caliper. The position for measuring the dimensions of the ALSs is shown in Figure 2. The morphology of the nontreated ALS and the treated ALSs samples were investigated using a field emission scanning electron microscope (Zeiss, Merlin compact). All specimens were coated with carbon by using a sputter coater before testing to create conductive conditions. The cross section and the surfaces of both sides of the nontreated ALS and the surfaces of the treated ALSs specimens were observed. Thermal degradation of the ALSs was determined under nitrogen atmosphere using a thermogravimetric analyzer (TGA, TGA8000, Perkin Elmer,

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USA). The temperature range was 30 to 500 °C and the heating rate was 5 °C/min [21].

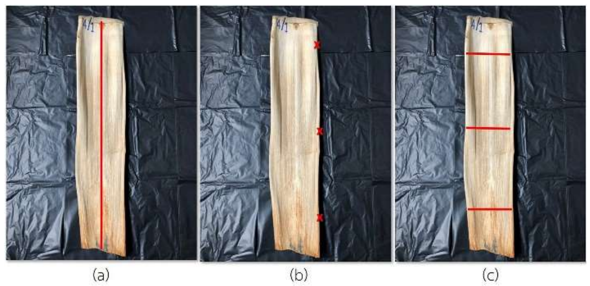

Figure 2 Positions for measuring the dimension of the ALSs: (a) length, (b) thickness, and (c) width.

2.4 Tensile testing of the ALSs

The tensile properties were analyzed in accordance with ASTM standard D3039/D3039M-08 (standard test method for tensile properties of polymer matrix composite materials) [22]. Specimens with a size of 200 mm x 20 mm x varying thickness were cut from the nontreated ALS and treated ALSs [19]. The specimens were tested at room temperature using a tensile testing machine (model Z010 Zwick Roell, Germany). The crosshead speed was 1 mm/min. At least five specimens were tested from each sample. The average values with standard deviation are reported.

3 Results and Discussion

3.1 Characterization of nontreated ALS

The average dimensions of the ALSs were obtained from 25 pieces of ALSs randomly collected from around the area of Nong Bua Sub-district, Ratsada, Trang, Thailand. The average length and width of the ALS were 30.5 ± 3.7 inches (77.5 ± 9.4 cm) and 7.6 ± 0.9 inches (19.3 ± 2.3 cm), respectively. The average thickness of the ALSs was 2.0 ± 0.4 μm. Junlacoupt and Srisuwan in [10] reported that the average length, width and thickness of the ALS were 66.2 cm, 23.0 cm and 2.5 μm, respectively. The SEM micrograph of the inner surface of the nontreated ALS in Figure 3(a) shows a rarely smooth surface. The outer surface of the nontreated ALS was rough (Figure 3(b)). Microscopic pores are visible in both the inner and the outer surface of the nontreated ALS. The average diameter of the micro-pores reported by Dissanayake, *et al.* [23] was approximately 4 μm. An SEM micrograph showing the cross-section area of the

ALS shows the xylem and phloem of the ALS (Figure 3(c)). The xylem are long tubes without a cross-wall with a length of >50 mm and an approximate diameter of 70 µm. The phloem are shorter tubes than the xylem. Their approximate length and diameter are 150 mm and 10 to 30 µm, respectively [9].

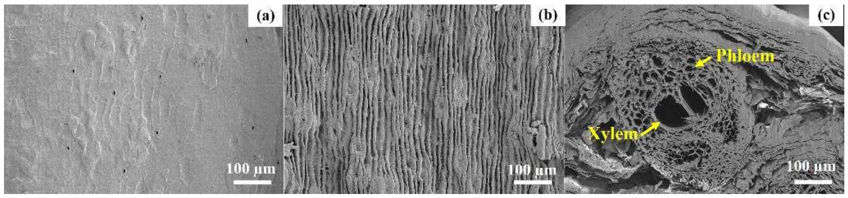

Figure 3 SEM micrographs of ALSs: (a) inner surface of ALS, (b) outer surface of ALS and (c) cross-section area of ALS.

3.2 Effect of Alkaline Treatment on Chemical Structure and Surface Morphology of ALSs

ALS is a lignocellulose consisting of α-cellulose, lignin, hemicellulose, pectin, and nonessential parts of fatty oils and wax, and aqueous extract [3,24]. Alkaline treatment is generally applied to cellulosic materials to reduce their hydrophilicity and to partially remove the hemicellulose and impurities such as waxes and oils on the surface [21]. After the alkaline treatment, the surface of the ALSs was characterized by FTIR. The FTIR spectra of the nontreated ALS compared with the NaOH-treated ALSs and the $NaHCO_3$-treated ALSs are shown in Figure 4(a) and (b), respectively. The spectrum of the nontreated ALS has peaks at 3747 cm^{-1} and 3326 cm^{-1} for hydrogen-bonded O–H stretching vibration [17,25]. The peaks at around 2919 cm^{-1} and 2849 cm^{-1} corresponded to the cellulose, hemicellulose, and lignin vibrations from the stretching of asymmetric and symmetric methyl and methylene C–H groups [19,26]. The peak at 1736 cm^{-1} corresponds to carbonyl C=O vibration, which extends crosslinks of ester and ether between cellulose and lignin or cellulose and hemicelluloses [19,23]. The peak at 1648 cm^{-1} is associated with H–O–H bending of absorbed water and C–H deformation of lignin. This peak was reduced by the alkali treatment because of the increase of amorphous cellulose at the expense of crystalline cellulose, which results in an improvement of the moisture resistance properties of the ALSs [19,27,28]. The peaks of C–O–C stretching and C–O ester bond stretching (glycosidic linkage) of cellulose and lignin were detected in the band around 900-1200 cm^{-1} [26]. The FTIR results show that the characteristic peaks at 3747 cm^{-1}, 1736 cm^{-1}, and 1648 cm^{-1} of the nontreated ALS were reduced after treatment with NaOH (Figure 4(a)) and $NaHCO_3$ (Figure 4(b)) due to the partial removal of cellulose, hemicellulose, and lignin components from the ALS surface. Moreover, the peaks of the alkaline treated ALSs decreased with an increase of the alkali concentration.

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The effects of the NaOH and $NaHCO_3$ treatments on the surface morphology of the ALSs are shown in Figure 5. The partial elimination of cellulose, hemicellulose, lignin, and nonessential parts produced a cleaner and rougher surface of the NaOH-treated ALSs (Figure 5 (b-d)) and a cleaner surface of the $NaHCO_3$-treated ALSs (Figure 5 (e-g)).

These results confirmed that cellulose, hemicellulose, and lignin were partially removed from the ALSs surface after chemical treatment with NaOH and $NaHCO_3$. This result is in accordance with previous studies by Ashok, *et al.* [17], Banagar, *et al.* [27], Nayak & Mohanty [28], and Shenoy, *et al.* [29]. In addition, the reduction of the intensity of the peak at 1648 cm^{-1} after the treatments with NaOH and $NaHCO_3$ increased the moisture resistance properties of the ALSs. An increase of the moisture resistance properties of ALSs after alkaline treatment may decrease or solve the problem of molding due to the humidity of ALSs in the storage step of ALS plate production.

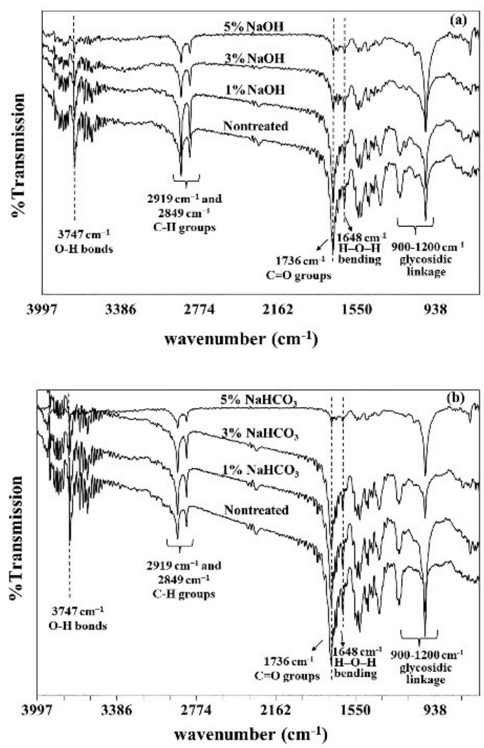

Figure 4 FTIR of nontreated ALS compared with (a) NaOH-treated ALSs and (b) $NaHCO_3$-treated ALSs at various concentrations.

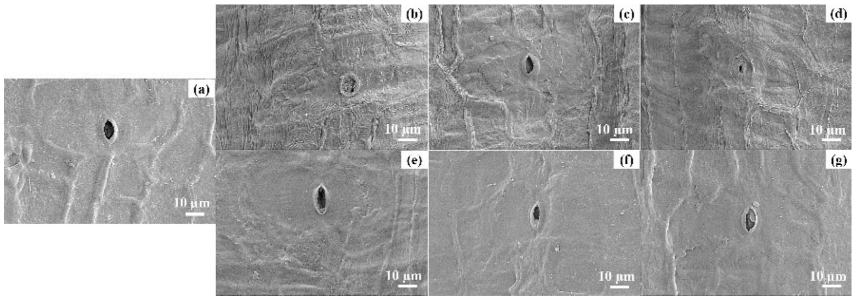

Figure 5 SEM micrographs of nontreated ALS and treated ALSs: (a) untreated ALS, (b) 1% NaOH, (c) 3% NaOH, (d) 5% NaOH, (e) 1% $NaHCO_3$, (f) 3% $NaHCO_3$, and (g) 5% $NaHCO_3$.mv

3.3 Effect of Alkaline Treatment on Thermal Degradation and Tensile Properties of ALSs

Differential thermogravimetry (DTG) and TGA thermograms of the ALS samples are shown in Figure 6 (a-d) and the thermal degradation temperatures (T_d) and the onset temperatures ($T_{d\ onset}$) of the different ALS samples are listed in Table 1.

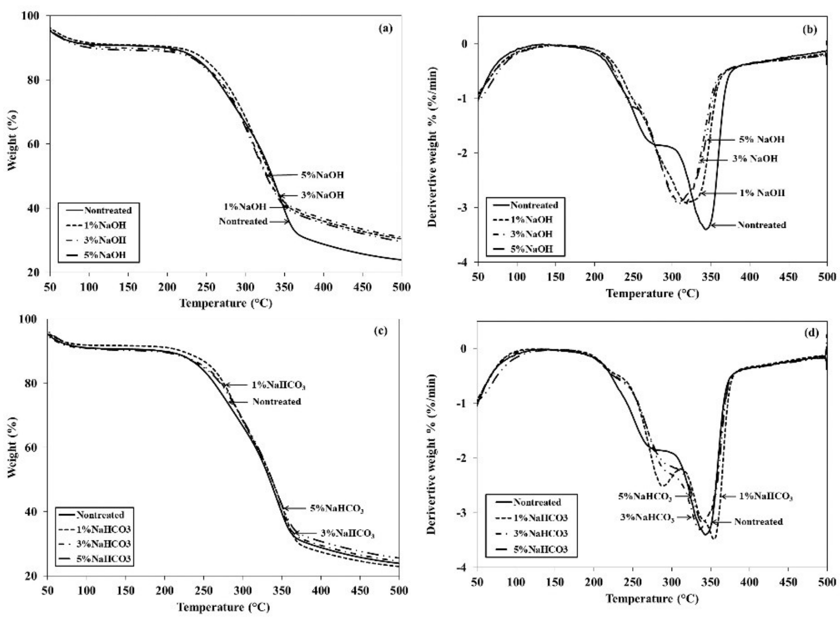

Figure 6 TGA and DTG thermograms of the nontreated ALS compared with the NaOH-treated ALSs ((a) and (b)) and $NaHCO_3$-treated ALSs ((c) and (d)).

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Table 1 Thermal degradation and tensile properties of the ALSs.

Sample name	Degradation temperature (°C)				Tensile properties		
	$T_{d\ onset\ 1}$	$T_{d\ onset\ 2}$	T_{d1}	T_{d2}	E (MPa)	σ_b (MPa)	ε_b (%)
Nontreated ALS	237	324	280	343	161 ± 7	21.6 ± 4.7	11.3 ± 0.9
1% NaOH	228	290	-	319	194 ± 22	30.3 ± 3.8	12.6 ± 2.0
3% NaOH	221	280	-	313	148 ± 20	34.6 ± 2.5	14.0 ± 1.2
5% NaOH	225	283	-	307	104 ±22	23.3 ± 7.7	11.3 ± 0.9
1% $NaHCO_3$	203	325	288	354	171 ± 19	19.3 ± 1.2	9.4 ± 0.6
3% $NaHCO_3$	204	323	298	336	130 ± 22	24.8 ± 4.3	12.2 ± 1.1
5% $NaHCO_3$	197	329	296	340	157 ± 28	26.4 ± 3.6	12.1 ± 0.6

T_d was the temperature at the peak of the DTG curve. $T_{d\ onset}$ was the temperature of degradation at the initial weight loss, obtained from the TGA curve. According to the TGA thermogram of the nontreated ALS, two $T_{d\ onset}$ were observed. $T_{d\ onset\ 1}$ was 237 °C and $T_{d\ onset\ 2}$ was 324 °C, attributed to the degradation of hemicellulose and cellulose, respectively [23]. The DTG thermogram of the T_{d1} and $T_{d\ 2}$ of the nontreated ALS shows small peaks at 280 °C and 343 °C, indicating the pyrolysis, decomposition and degradation of hemicellulose and cellulose, respectively [24]. A reduction of the degradation temperature of the ALSs was found after the surface treatment with NaOH. The $T_{d\ onset}$ and T_d of the ALSs slightly decreased with an increase of the NaOH concentration. The surface treatment with $NaHCO_3$ slightly affected the thermal stability of the ALSs.

The effect of type and concentration of the solution of chemicals ALSs used in the surface treatment on the tensile properties of the ALSs are shown in Figure 7 and Table 1. It was found that the overall tensile properties of the ALS treated with NaOH were better than those of the ALS treated with $NaHCO_3$.

The surface treatment with 1% of both NaOH and $NaHCO_3$ increased the tensile modulus of nontreated ALS, while the tensile modulus of the nontreated ALS decreased with an increase of both the NaOH and the $NaHCO_3$ concentration, at 3% and 5% (Figure 7 (a)).

The surface treatment with 1% and 3% of NaOH increased the tensile stress of the ALSs as compared with the nontreated ALS. The surface treatment with $NaHCO_3$ slightly changed the tensile stress of the ALSs (Figure 7 (b)) after treatment with 1% NaOH, while the tensile strain slightly changed. In addition, the surface treatment with NaOH and $NaHCO_3$ slightly changed the % tensile strain of the ALSs (Figure 7 (c)).

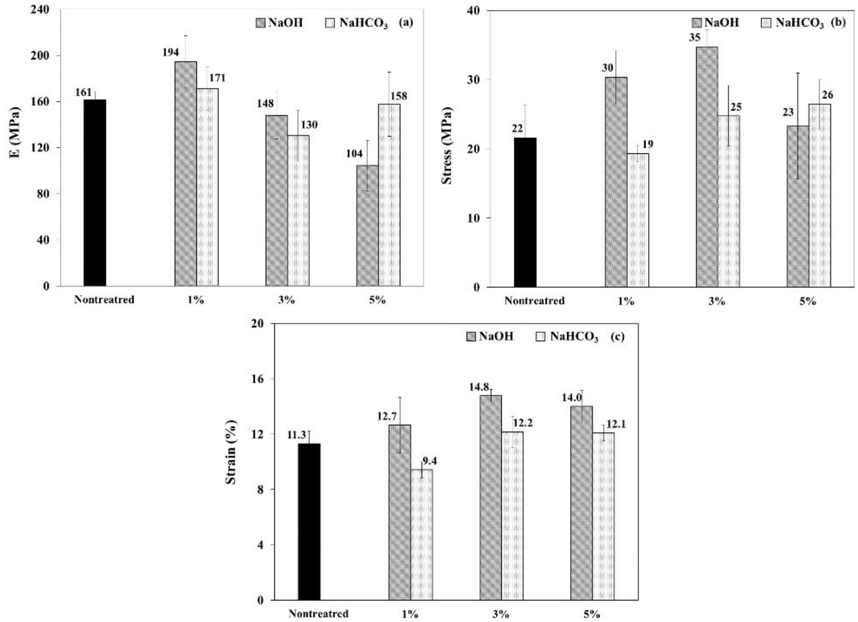

Figure 7 Effect of alkaline treatment on tensile properties of the ALSs: (a) tensile modulus, (b) tensile stress and (c) tensile strain.

4 Conclusions

Alkaline treatments were applied to ALSs, after which the surface modification of the ALSs was characterized by FTIR and SEM. The thermal stability and mechanical properties of the ALSs was studied through TGA and tensile testing, respectively. Surface treatment with NaOH and $NaHCO_3$ partially removed hemicellulose, cellulose, lignin, and impurities from the surface of the ALSs and could improve the moisture resistance properties of the ALSs, as confirmed by FTIR. The SEM and TGA results showed that surface treatment with NaOH made the ALSs' surface rougher, and the decomposition temperature was slightly reduced. Surface treatment with $NaHCO_3$ had no effect on the surface of the ALSs but it did slightly change the decomposition temperature. The overall tensile properties of the ALSs treated with NaOH were better than those of the ALSs- treated with $NaHCO_3$.

The surface treatment with 1% NaOH and 1% $NaHCO_3$ increased the tensile modulus of the nontreated ALS. The 1% and 3% NaOH-treated ALSs showed higher tensile stress than the nontreated ALS. The tensile strain slightly changed after surface treatment. The results showed that surface treatment with NaOH and $NaHCO_3$ could enhance the moisture resistance properties of the ALSs and did

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not impair the thermal decomposition properties and tensile properties of the ALSs. Alkaline treatment with NaOH and $NaHCO_3$ would be a very good solution for solving the problem of molding due to moisture during storage of ALSs before disposable ALS plate production. Alkaline treatment with 1% NaOH and 1% $NaHCO_3$ are the optimum conditions for ALS plate production. However, alkaline-treated ALS plate production and more studies on factors such as dimension stability, density, and water absorption still need to be done.

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