

Effect of Moisture Content on the Tensile Properties of Areca Leaf Sheath

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ABSTRACT

The growing environmental concerns regarding non-biodegradable disposable dining ware call for sustainable alternatives. Fallen Areca leaf sheath (ALS) presents a promising solution, as it is an eco-friendly, biodegradable material that can be molded into disposable products through a simple heat pressing process. Before heat pressing, ALS is immersed in water to enhance its pliability. However, the quality of ALS-based products is highly influenced by moisture content, which affects the material's tensile properties during the molding process. This study investigates the effect of moisture content on the tensile properties of ALS to determine the optimal soaking duration. It was found that as the moisture content increases, the tensile strength of ALS decreases when soaked for less than 5 minutes. Meanwhile, the elongation at break improves after soaking. Water acts as an effective plasticizer for ALS, enhancing its ductility for the heat pressing process. These findings provide valuable data for manufacturers, allowing for more consistent product quality and potentially reducing waste in the production of disposable dining ware.

Keywords: *Areca leaf sheath, Disposable, Lignocellulosic material, Tensile properties, Soaking time*

1. INTRODUCTION

The existence of disposable packaging and dining wares has long been a controversial topic among nature lovers. In such time where nature preservation has become unarguably one of the main priorities in our world today, research on bio-degradable disposable dining ware should be emphasized to provide more environmentally friendly alternatives. One of the promising alternatives is the Areca leaf sheath (ALS) disposables [1-3].

For decades in India, the fallen Areca leaf sheath, from Areca palm (scientifically known as Areca Catechu Linnaeus) has been used to make disposable plates and bowls, as an eco-friendly disposable product [1-3]. This unusable fallen leaf sheath can be manufactured through simple heat pressing process to deform it into the shapes of the molds [2,4]. Firstly, the fallen ALS is collected and sun dried before being stored up. The ALS can be stored for up to 12 months. Then, before the ALS is heat pressed into different structures, it is washed, and then soaked to gain some moisture for better pliability during heat pressing process [4-5].

Moisture content (MC) plays a major role in influencing the mechanical performance of ALS to produce disposable products of good quality [6-7]. ALS as a lignocellulosic material is hydrophilic in nature [8-9]. Hydration of ALS makes the material more flexible and compliant for the subsequent molding process [10].

However, excessive moisture in ALS might induce swelling and excessive plasticizing effect which leads to dimensional instability [6,11-12]. On the other hand, ALS

with low MC (below 5%) causes the material to crack easily during heat pressing process [6].

Ductility is a measure of a material's ability to deform plastically without fracturing. It is an important property for materials that are subjected to heat pressing, as it allows the material to deform into the desired shape without cracking or breaking [13]. Meanwhile, moisture content can significantly affect the ductility of a material. Several studies have demonstrated that increasing the moisture content of natural fiber-reinforced polymer composites improved their ductility and toughness [14-16]. Therefore, the effect of MC of ALS on its tensile properties should be investigated for better mechanical performance during heat pressing process. This study provides a practical database and process guideline for the industrial application of ALS in the production of disposable dining ware, which has the potential to significantly reduce defective products and increase industry profits.

This study investigates the effect of soaking time on the tensile properties of ALS. The results of the tensile tests were analyzed using Analysis of Variance (ANOVA) and Tukey Pairwise comparison methods in statistical analysis software, Minitab 18.

2. MATERIAL AND METHODS

2.1. Material

ALS samples originated from Indonesia (Arecaceae Catechu L.) supplied by Alora Eco Green Products Sdn. Bhd. were used in this study. The samples were stored in an air-conditioned laboratory (24°C, 65% humidity) after collecting. Prior to the experiment, the surface of the samples was cleaned thoroughly with a wet cloth to remove the particles accumulated. The ALS has non-uniform thicknesses over the surface. Maximum and minimum thickness measured by using a Vernier caliper (0.05 mm precision) from the ALS is 8.2 mm and 0.9 mm respectively. Figure 1 illustrates the different thicknesses along the ALS in cross section.

Due to such huge difference between the thicknesses, the minimum and maximum thicknesses used for this study were 1 mm and 4 mm respectively. The ALS parts with thicknesses above 4 mm are difficult to cut into sample sizes manually. Whereas the thicknesses below 1 mm were observed to be very fragile. These abandoned parts constitute only a very small portion of the whole surface area of ALS.

Due to the concavity of ALS in the center, the ALS are to be flattened using a vertical laboratory padding mangle. Previous study shows that flattening the ALS will improve its tensile strength significantly [17-18].

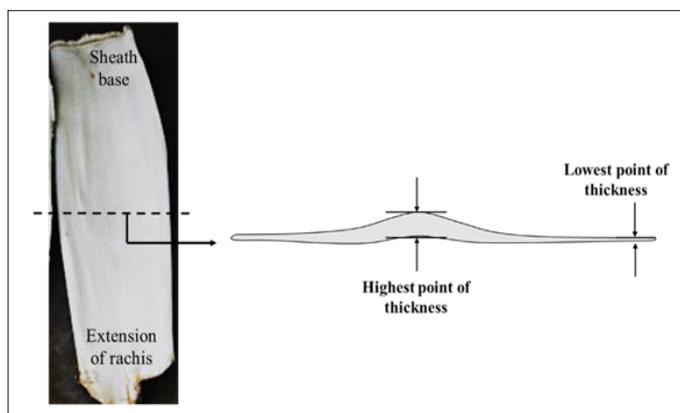


Figure 1. The ALS in cross section view.

2.2. Moisture Content

Oven drying method is employed for determination of MC of ALS, due to its simplicity to conduct and reliable results produced. The samples were dried at the temperature of $105 \pm 2^\circ\text{C}$ for 2 hours in accordance with ASTM D4442-92 (Reapproved 2003). The methods described in this standard refer to the oven-dry basis. Because oven-dry mass is used, MC values may exceed 100%. The type of oven used to dry the samples is a convection oven (Memmert D06062 Model 600, German). ALS samples were cut to the convenient size that can fit onto the weighing balance base then measured their weights before and after dried, with 0.01 g precision.

2.3. Preliminary Study

There were 8 levels of soaking time selected, which are 1 min, 2 min, 3 min, 5 min, 10 min, 15 min, 20 min and 25 min, with 10 replications for each level. The ALS samples were prepared by first flattening them under 5 bar pressure, and then cut into the size of 5 cm \times 5 cm [17]. The short time interval decided for the first 3 parameters (1 min, 2 min, and 3 min) is due to a common hydrophilic behavior by lignocellulosic materials where water absorption rate is rapid at the early stage of soaking before they are slowly saturated with water [10,19-20]. It is crucial to verify this behavior in ALS.

After the ALS were soaked in water, they were dried in an oven for 2 min at 105°C to remove excessive water on the ALS surface. Next, the weight of ALS samples is recorded, before drying the samples to oven-dried weight to calculate for its MC in accordance to ASTM D4442-92.

2.4. Tensile Test

Tensile tests were conducted to evaluate the ultimate tensile strength (UTS) and elongation at break (EB) of ALS. The tensile tests were conducted in accordance with ASTM D638-14, Standard Test Method for Tensile Properties of Plastics. The tests were conducted on Universal Testing Machine (UTM) (Lloyd LR30K Plus, Canada), which was connected to a computer with testing software Nexygen 4.1 installed to calculate the tensile responses. The stress-strain curves were recorded. The cross-head speed was set at 5 mm/min as specified in the standard, for a rupture time within 5 min. The load cell used has a capacity of 5 kN. The calculations for tensile properties were done in the computer automatically.

The ALS were cut manually into dumbbell-shaped samples, the dimensions are as illustrated in Figure 2. The samples that break outside of the narrow gauge length section were discarded and new samples were taken for retests. Five best samples for each group that break within the gauge length of 50 mm were obtained for result analysis. The width and thickness of the ALS samples along their gauge length were measured with a Vernier caliper to the precision of 0.05 mm. The measurement for thickness was taken at 3 different points within the gauge length to obtain an averaged thickness value.

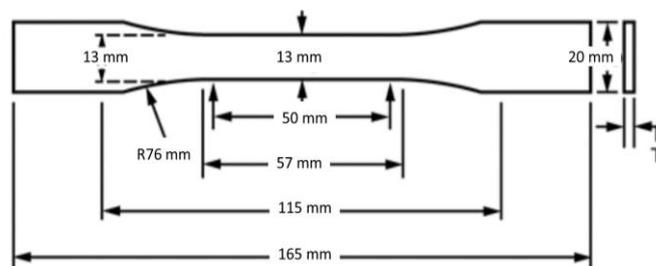


Figure 2. Dimension of tensile test samples.

The purpose of tensile tests is to determine whether the UTS and EB of ALS are significantly different at different levels of soaking time. There are 8 levels of water soaking time investigated, (1 min, 2 min, 3 min, 5 min, 10 min, 15 min, 20 min, and 25 min).

In accordance to the requirement of ASTM D638-14, each sample group has 5 replications, which made up to a total of 40 observations for each grain direction. 2 sets of experiments were conducted for ALS in both grain direction and transverse direction.

Then, the samples were mixed and classified into different soaking time groups randomly. The categorized samples were then submerged into water group by group. After each group of samples was soaked, the samples were oven dried for 2 min at 105°C to remove excessive water on the surface. Next, tensile testing was conducted on the samples immediately. While a sample is undergoing the tensile testing, others were kept in a closed container to minimize moisture loss to the surrounding. After a group of samples were tested, the processes of soaking and oven drying were again started for the next group of samples consecutively. The data obtained from tensile tests were analyzed statistically afterward. Figure 3 illustrates the overall process flow of experimental design.

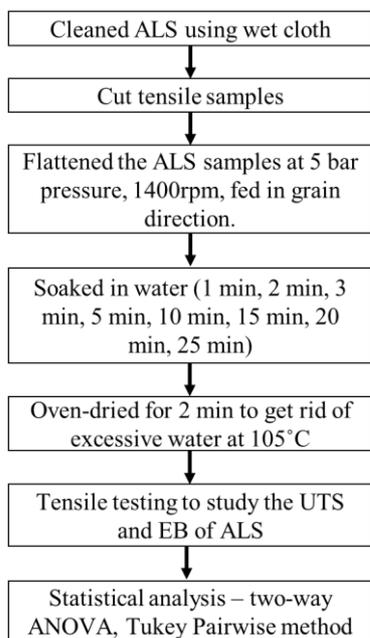


Figure 3. Process flow chart of experimental design.

2.5. Statistical Analysis

The statistical analysis category adopted for current study is the inferential statistics. The techniques used are the ANOVA and the Tukey's Comparison method. These techniques are used due to their abilities to compare two or more groups of sample, to determine whether there is significant difference within or between the sample groups. The analysis process was carried out using statistical analysis software, Minitab 18. The values of sum

of squares (SS), mean squares (MS), F-values and p-values are automatically calculated and generated by Minitab 18.

3. RESULTS AND DISCUSSION

Water soaking time is a very important factor in manipulating the MC of ALS. The preliminary study explores the relationship of ALS soaking time with its MC.

The ALS achieved 29.10% MC after soaking for 5 min. ALS should obtain MC below 30% before heat press to avoid swelling, thus losing its rigidity [6]. It can be seen that as the soaking time increased, the MC of ALS samples also increased. The graph plotted in Figure 4 further illustrates this trend.

It is noticed that, the standard deviation values for MC of ALS for soaking time at 1 min, 2 min and 3 min are very low compared to other groups. Apart from the complex structure of ALS, this might occur due to the fact that water saturation in ALS after soaked up to 3 min was still very low, and it is below the Fiber Saturation Point (FSP).

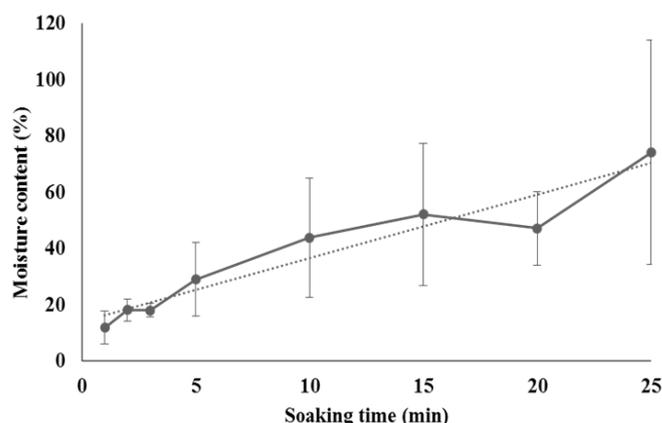


Figure 4. MC versus soaking time for tensile samples.

According to Bajpai, FSP of wood as a lignocellulosic material occurs at around 25 - 30% MC [21]. The ALS soaked for 5 min gave an average of 29.10% of MC, which is within this range. And it is interesting that the standard deviation values started to increase from 5 min sample group up until 25 min, indicating high variability in term of MC in ALS. The author deduces that ALS probably reaches its FSP when it is soaked for 5 min. The high variability of MC above 30% is probably caused by the unstable free water in the ALS, resulting in unstable weight gain of ALS for MC calculation.

At MC below FSP, the water molecules are chemically adsorbed through hydrogen bonding within ALS matrix (mainly on hemicellulose), known as bound water [10,21-22]. Whereas at MC above FSP, water is accommodated in the empty pores of ALS as free water through a process called capillary absorption process [10,21-22]. The chemically adsorbed water molecules are energetically more stable, and they require higher energy to be removed from ALS compared to the free water [21,23]. The high

variability of ALS at MC above 30% is probably caused by the unstable free water in the ALS, resulting in unstable weight gain of ALS for MC calculation.

Despite the widely dispersed data points, the ANOVA results as shown in Table 1 concluded that soaking time is significantly affecting the MC of ALS, with a p-value of 0.00. The result of Tukey’s method in Table 2 indicates that the soaking time of 1 min, 2 min, 3 min, and 5 min are not significantly different from one another. The soaking times starting at 15 min until 25 min yielded significantly different MC than soaking time from 1 min to 10 min. As a summary, in accordance to the suggestion by Kalita *et al.*, ALS is to be soaked for maximum 5 min to achieve 30% MC [6].

Table 1 ANOVA result: effect of soaking time on MC of ALS samples for tensile testing

Source	df	Adj SS	Adj MS	F-value	p-value
Soaking time	7	31583	4511.80	11.75	0.00
Error	72	27648	384		
Total	79	59231			

Table 2 Grouping information by Tukey’s method: effect of soaking time on MC of ALS

Pressure (bar)	N	Mean (%)	Grouping			
25	10	74.20	A			
15	10	52.51	A	B		
20	10	47.23	A	B		
10	10	43.89		B	C	
5	10	29.10		B	C	D
2	10	18.26			C	D
3	10	18.15			C	D
1	10	12.00				D

3.1. Effect of Moisture Content on Tensile Properties

The data collected from tensile testing is summarized in Tables 3 and Table 4. There are huge differences on the UTS and EB values between grain and transverse directions due to anisotropic characteristics of ALS [24-25]. The UTS values of samples in grain direction are greater than transverse direction ones; while the EB values of the samples in grain direction are less than the transverse direction samples. Such results are similar to the findings of previous studies [17-18].

In terms of UTS for grain direction samples, a rapid drop of values can be observed for the soaking time from 1 min to 5 min, before the UTS values remained almost constant until 25 min of soaking. On the other hand, the UTS values

for transverse samples do not show great fluctuation, and there is no significant trend observed.

On the other hand, there is no consistent pattern for EB values for grain direction samples. However, it can be said that as the soaking time increases, the values of EB generally increase. This phenomenon is observed from 1 min until 5 min of soaking time. Further moisture intake afterward seems to cause negligible changes. More investigations are done to find out the significance of soaking time using statistical analysis techniques. Unlike the EB results from grain direction samples, the transverse direction ALS samples show great fluctuations in the data collected.

Besides, the stress-strain curves of ALS samples for different grain directions portrayed different tensile behaviors as displayed in Figure 5 and Figure 6.

Table 3 UTS and EB for grain direction ALS

Soaking time (min)	Grain direction	
	UTS (MPa)	EB (%)
1	31.00 ± 2.30	6.79 ± 6.23
2	21.97 ± 6.66	7.86 ± 0.41
3	19.32 ± 2.30	11.10 ± 6.23
5	16.57 ± 4.25	12.12 ± 6.73
10	14.93 ± 5.98	9.61 ± 3.08
15	12.56 ± 3.80	13.04 ± 5.44
20	13.11 ± 2.00	9.50 ± 4.76
25	14.24 ± 2.62	10.38 ± 5.12

Table 4 UTS and EB for transverse direction ALS

Soaking time (min)	Transverse direction	
	UTS (MPa)	EB (%)
1	2.91 ± 1.93	38.82 ± 11.82
2	1.87 ± 0.67	75.93 ± 15.89
3	2.78 ± 1.06	76.51 ± 12.44
5	1.45 ± 0.43	42.28 ± 16.68
10	2.31 ± 0.46	33.53 ± 7.97
15	1.69 ± 0.28	70.92 ± 14.50
20	1.73 ± 0.32	78.83 ± 20.99
25	1.68 ± 0.15	42.90 ± 19.16

The graphs of grain direction samples appeared smooth, while those of the transverse direction samples appeared rough. This shows that the samples in grain direction were constantly elongating under the loading of the tension force. In the process, the fibers tend to align themselves from their original helical structure into parallel to the stress axis to resist the tension [26-27].

This realignment creates the non-linear tensile behavior for grain direction samples [27]. When a high maximum stress is reached, the first single fiber started rupturing

until the whole sample failed completely in split second under the high stress. Such behavior agrees with the description of tensile behavior of hemp fiber yarns [28]. Whereas, the irregular rough graph offered by samples in transverse direction suggests that the layer delamination of ALS samples happened in stages instead of in an instance. The moment after a layer is ruptured gave a temporary stress reduction to the sample, before the next new layer carried the tension stress again. This relaxation and tension cycle went on constantly causing irregular stresses applied on the sample as it was elongated before failing completely. This is a favorable characteristic as it increases the elongation of ALS significantly, contributing to its ability to deform more before cracking at macroscopic level.

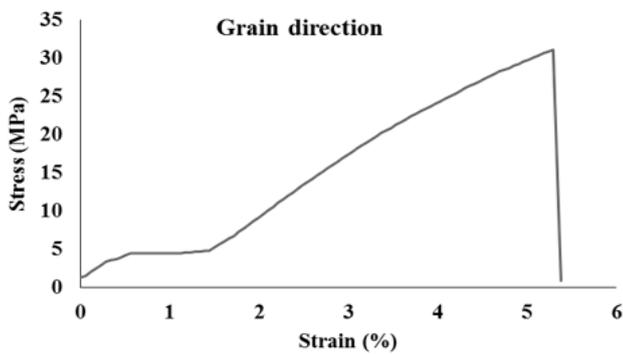


Figure 5. Stress-strain curves of ALS in grain direction for 1 min soaking time.

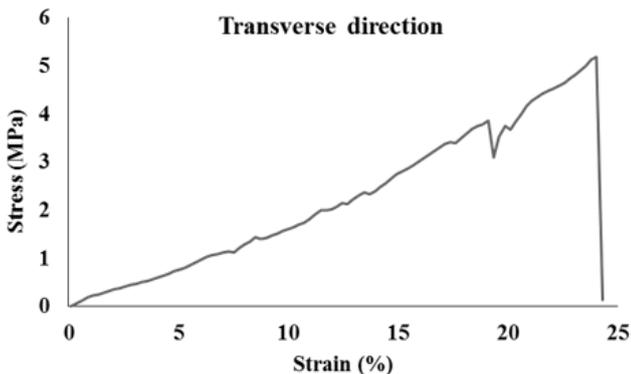


Figure 6. Stress-strain curves of ALS in transverse direction for 1 min soaking time.

From Figure 7, the maximum UTS value is obtained at 1 min soaking group. Referring to Table 4, the UTS obtained from 1 min soaking is 31.00 MPa, which is only 2.71% lower than the highest value (31.86 MPa) produced by dry ALS flattened at 5 bar in previous study [17].

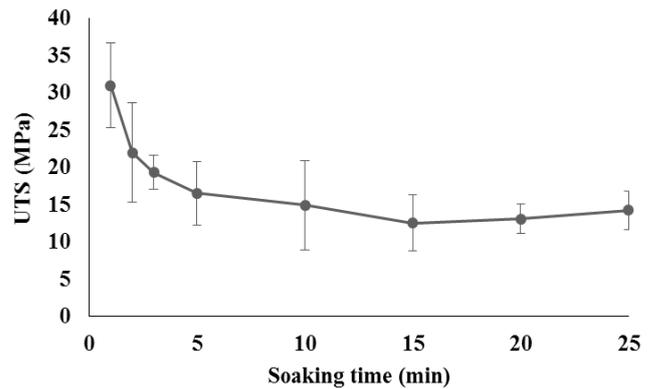


Figure 7. UTS of ALS grain direction against water soaking time.

From the graph, the UTS of ALS decreased sharply from 1 min until 5 min, before it remained almost constant for the rest of the sample groups. It is deduced that after soaking for 5 min, the ALS samples had reached the FSP. The tensile properties of ALS would not be affected significantly by MC beyond the FSP [26]. Thus, it can be said that the UTS of ALS in longitudinal grain direction decreases as the soaking time increases.

From the ANOVA results shown in Table 5, the soaking time is confirmed to have significant influence towards the UTS of ALS, as the p-value produced is 0.000.

Table 5 ANOVA result: effect of soaking time on UTS of grain direction ALS

Source	df	Adj SS	Adj MS	F-value	p-value
Soaking time	7	1327.40	189.62	7.54	0.00
Error	32	804.40	25.14		
Total	39	2131.80			

To probe further, the Tukey method comparison results in Table 6 show that the result from 1 min soaking time is significantly different from all other soaking time groups except for 2 min sample group. Thus, it can now be confirmed that as the MC of ALS increases, its UTS decreases. This finding is in alignment with a number of research, where the tensile stress is inversely proportional to the MC of bamboo samples at grain direction [25,30-31]. The reason that a higher MC causes negative impact to the strength of ALS in grain direction is because moisture swells the cell wall of the lignocellulosic material [19,32].

Table 6 Grouping information by Tukey method: effect of soaking time for grain direction samples on UTS

Soaking time (min)	N	Mean (%)	Grouping	
1	5	30.99	A	
2	5	21.97	A	B
3	5	19.32		B
5	5	16.57		B
10	5	14.93		B
25	5	14.23		B
20	5	13.11		B
15	5	12.56		B

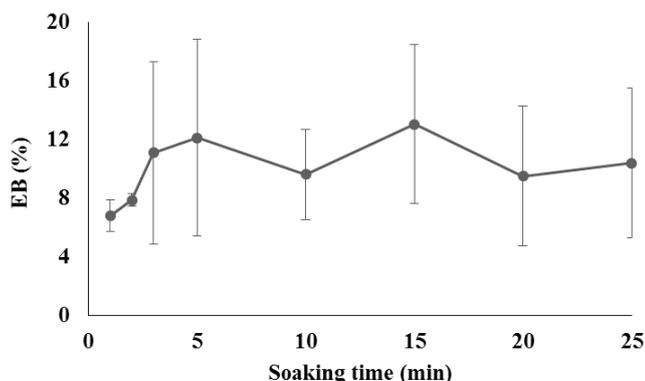


Figure 8. EB of ALS in grain direction against water soaking time.

The MC impacts the EB of ALS differently than its UTS. Although there is no regular trend produced after soaking time of 5 min, the water absorbed by ALS improved its ability to elongate before fracture when it was soaked for 1 min until 5 min. This is a result of plasticizing effect of moisture in ALS, giving it more flexibility. After a maximum point of MC is reached, the plasticizing effect will be reduced, thus causing little effect on the tensile properties of ALS [11,29].

The ANOVA result in Table 7 reports a p-value of 0.594, indicating that soaking time does not play a significant role in manipulating the EB of ALS. Such conclusion is made upon the irregular fluctuations of nearly similar values from soaking time of 10 min until 25 min. It is not completely true as the unique plasticizing effect that moisture applied on ALS below 5 min soaking time has shown an obvious increasing trend.

During water immersion, the water molecules force themselves between the micro-fibril chains of ALS and push the chains apart from each other [11]. The moisture in ALS acts as plasticizer for ALS, which decreased its tensile strength and rigidity [11,33].

When it comes to EB of ALS in grain direction, the highest value of EB is found in the group that was soaked for 15 min, that is 13.04% (refer to Table 3). This value is 36.56% higher than the maximum value produced by dry ALS samples that were pressed at 5 bar, which is 8.27% [17]. From Figure 8, it is observed that there is no consistent pattern for EB values. However, there is a constant increase of EB from 1 min to 5 min sample groups, before the EB values fluctuated inconsistently afterward.

Table 7 One-way ANOVA result: effect of soaking time on EB of grain direction ALS

Source	df	Adj SS	Adj MS	F-value	p-value
Soaking time	7	151.70	21.66	0.80	0.59
Error	32	867.10	27.10		
Total	39	1018.70			

The graph of UTS of ALS in transverse direction versus soaking time is illustrated in Figure 9. There is no consistent pattern shown in the graph. Unlike the pattern for ALS in grain direction, it is not possible to conclude that the UTS value decreased as the soaking increased. The maximum UTS value obtained after soaking is 2.910 MPa (Table 4), from sample group of 1 min soaking.

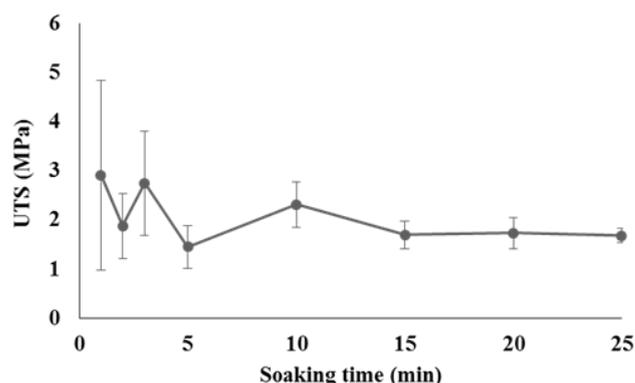


Figure 9. EB of ALS in transverse direction against water soaking time.

Based on the pattern of the graph, it can be said that, moisture absorption for 1 to 3 min has effectively softened the ALS for higher ductility. Whereas, further moisture absorption beyond 3 min might have caused the water molecules to be saturated between the ALS fibers, leading to larger dimensional expansion of ALS samples in transverse direction [21]. The hydrophilic characteristics of ALS contributed to more water penetrating into the micro-cracks existed in the samples, which may have increased the swelling stresses in ALS, thus accelerated the

failure of ALS samples [34]. However, due to the complex microstructure of ALS as organic material, the sample groups of 15 and 20 min soaking time managed to sustain longer under the stresses. Therefore, the highly unpredictable and irregular nature of ALS as a lignocellulosic material contributed to the variation in the result as well [19].

Interestingly, the drastic fluctuations of EB values of ALS in transverse direction led ANOVA to conclude that soaking time is a significant factor to influence the variation of EB values, as displayed in Table 8. The p-value obtained is 0.00, which shows a definite confidence that soaking time contributed massively to the variation of EB values. It is deduced that ANOVA considered the possibility that this data set portrays a quadratic model instead of a common linear model, and this hypothesis needs further investigation. The Tukey's grouping information is presented in Table 9 as reference.

Table 8 ANOVA result: effect of soaking time on EB of transverse direction ALS samples

Source	df	Adj SS	Adj MS	F-value	p-value
Soaking time	7	13523	1931.80	6.48	0.00
Error	32	9535	298.00		
Total	39	23058			

Table 9 Grouping information by Tukey method: effect of soaking time for transverse direction samples on EB

Soaking time (min)	N	Mean (%)	Grouping			
20	5	78.80	A			
3	5	76.51	A	B		
2	5	75.93	A	B		
15	5	70.92	A	B	C	
25	5	42.90		B	C	D
5	5	42.28		B	C	D
1	5	38.82			C	D
10	5	33.53				D

Ideally, the soaking time should be shortened to save time and cost of the industries. Therefore, a soaking time below 5 min can be considered.

4. CONCLUSION

To conclude, the tensile properties of ALS are unquestionably affected by the MC especially in grain direction, but to a certain extent. The UTS of ALS in grain direction is decreased, and the EB increased for soaking time before 5 min. Further soaking beyond 5 min yielded inconsistent or negligible changes to the tensile properties. The MC achieved after soaking for 5 min is probably the

FSP of ALS. The effect of MC on ALS in transverse direction does not exhibit uniform trend as ALS in grain direction. However, the absorbed moisture increased both the UTS and EB values generally compared to dry ALS. Therefore, water is an effective plasticizer to soften and increase the pliability of ALS for heat pressing process.

ACKNOWLEDGMENTS

Communication of this research is made possible through monetary assistance by Universiti Tun Hussein Onn Malaysia, the UTHM Publisher's Office via Publication Fund E15216.

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