

Patrick Chester, Master's Candidate

Dr. William Jordan, Advisor

Baylor University Department of Mechanical Engineering

ASEE-GSW 2016

**Effects of Chemical Treatments on the Properties of Individual Banana Pseudo-stem  
Fibers for use in LDPE composites**

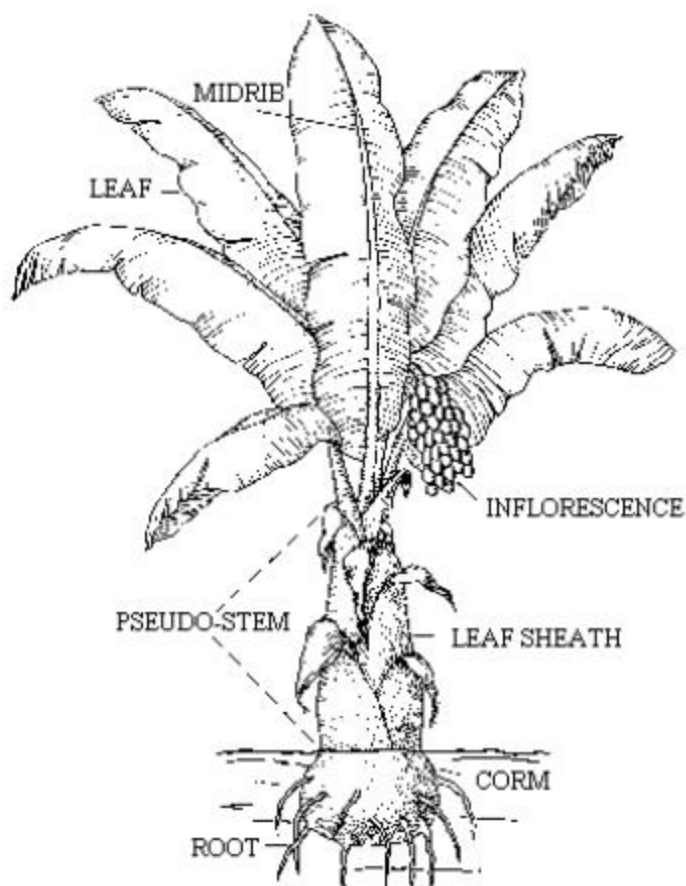
*Abstract*

For this study, banana pseudo-stem fibers were chosen as a possible natural reinforcement in low density polyethylene (LDPE). There are some challenges associated with using plant fibers in a polymer matrix, as the fibers naturally tend to absorb moisture. This leads to weakened fiber strength as well as poor bonding between the fibers and the LDPE matrix. Fortunately, there are some known ways to help improve fiber properties through chemical treatments which serve to decrease the hydrophilicity and/or improve the bonding between fiber and matrix. Two of such treatments, peroxide treatment and permanganate treatment, were studied for their effects on the properties of individual banana pseudo-stem fibers, taking into account various processing factors such as wetting, drying, and idle time in addition to basic tensile properties. It is shown that neither treatment appears to have a detrimental impact on the thermal resilience of the fibers. Peroxide treatment appears to enhance the fiber's overall strength, whereas permanganate treatment does not have a definite trend on the fiber's overall strength.

*Introduction*

In an effort to make lightweight, environmentally- friendly materials, there has been a push in industry and research to develop natural composites, or composite materials that utilize

renewable resources. Several natural fibers have been previously studied for their possible uses as natural reinforcement, including jute, sisal, palm, hemp, and flax [1]. One type of fiber that has been relatively unstudied is the banana pseudo-stem, especially in regards to LDPE composites. A location of pseudo-stem on the banana plant is shown in Figure 1.



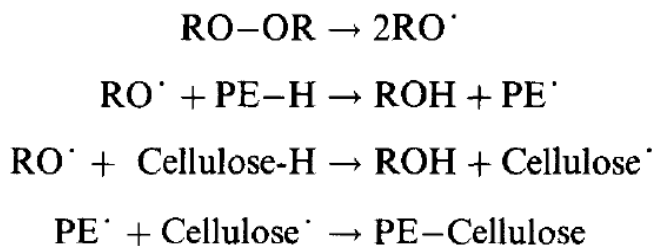
**Figure 1: Detailed Drawing of Banana Plant [2]**

Banana pseudo-stem fibers are very similar in composition to other natural fibers used as reinforcement, and thus could potentially have similar success in being implemented. There is an additional benefit to using pseudo-stem in that it is largely seen as an agricultural waste product, and thus finding a use for them in natural composites would have far-reaching benefits within the banana industry.

Like other natural fibers, there are some challenges associated with using banana pseudo-stem fibers in LDPE. Banana pseudo-stem fibers are naturally hydrophilic, whereas LDPE is hydrophobic. These incompatible properties lead to reduced bonding strength between the fiber and the matrix, lowering the composite material's overall strength. This problem can be alleviated, however, using chemical treatments or additives that serve to increase the bonding between fiber and matrix chemically (by introducing a mutually compatible bonding agent) or mechanically (by roughening the surface of the fiber).

One of the first people to study the effects of chemical treatments on banana fiber-LDPE composites was Joshua Weed [3]. In his research, he looked at 3 common treatments: alkali treatment, silane treatment, and the additive maleic anhydride. In addition to quantifying the overall effects of the treatments, Weed also studied the treatments' effect on the fibers by themselves. As a successor to Weed's work, this study will follow the same example of studying additional treatments' effect on the properties of the pseudo-stem fibers by themselves.

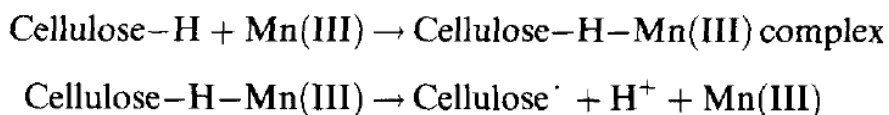
Joseph et al. [4] provides the blueprint for the additional treatments used in this study. He studied the effects of several chemical treatments on sisal-LDPE composites. One of these treatments was peroxide treatment. This treatment utilizes the "peroxide initiated free radical reaction" to increase the bonding between the fiber and matrix. The chemical reaction is shown below:



**Figure 2: Peroxide Treatment as described by Joseph et al. [4]**

Another treatment studied by Joseph et al. was permanganate treatment. This treatment utilized "The highly reactive  $\text{Mn}^{3+}$  ions [which] are responsible for initiating graft

copolymerization.” Graft copolymerization is a form of cross-linking which make it harder for polymer chains to move past each other, increasing the bonding strength. The chemical reaction for this process is shown below:



**Figure 3: Permanganate Treatment as described by Joseph et al. [4]**

Over the course of his work, Weed ran into some additional problems in the processing of his composite material. As he extruded the newly mixed banana fiber-LDPE composite material, he had it quickly cooled in a water bath. This allowed for the fibers to absorb some of the water. As he once again melted the material to make tensile test bars, the water trapped inside the fibers suddenly bubbled out as steam, leading to rapid expansion of the material as shown in Figure 4. This would leave voids within the material, compromising its strength. This effect was minimized through several cycles of heating and cooling (without water bath). He did express concern, however, that such methods may have thermally degraded the fibers [3].



**Figure 4: Rapid Expansion of composite material due to moisture content [3]**

In order to address this possibility, several thermal studies will be performed on untreated and treated fibers in order to determine things such as a safe operating temperature range, an optimal drying temperature, the effects of wetting and drying, and how these properties change over time.

## *Experimental Method*

### **Chemical Treatments**

The procedures for the chemical treatment of the fibers follow the method described by Joseph et al. very closely [4]. For the peroxide treatment, untreated fibers are soaked in a 6% solution of dicumyl peroxide in acetone for 30 minutes. The fibers were then decanted and allowed to air dry. For the permanganate treatment, untreated fibers were soaked in a solution of potassium permanganate in acetone (concentration varying from 0.005% to 0.2%) for 1 minute. The fibers were then decanted and allowed to air dry.

Once the treatments were complete, fibers were stored in a temperature-humidity control chamber. This was to ensure that all fibers started with the same moisture content.

### **Thermal Testing**

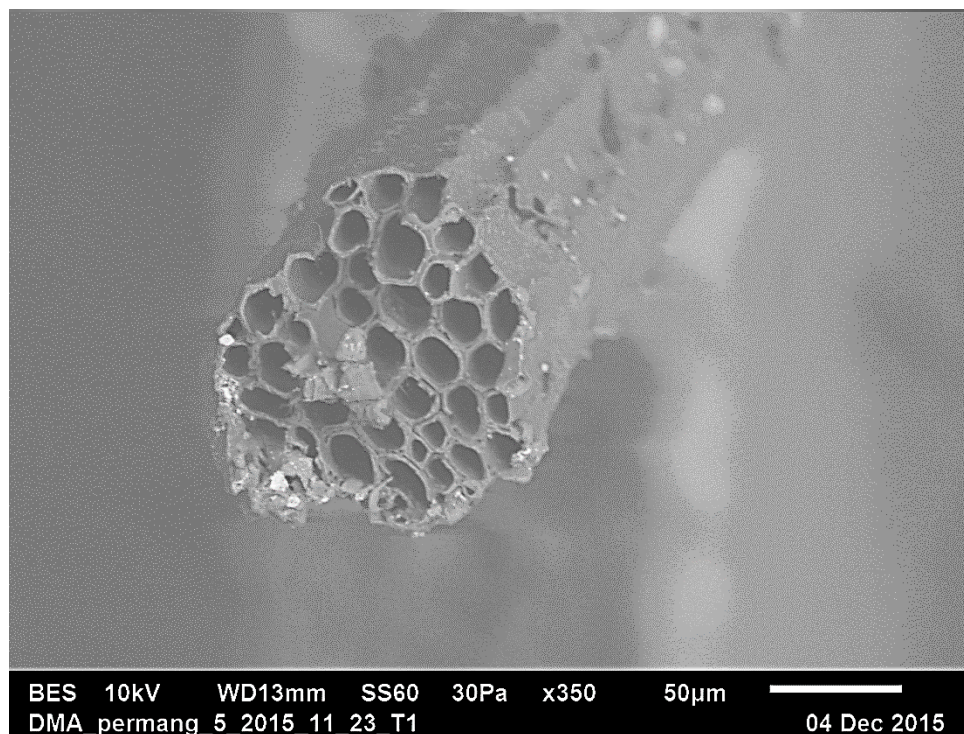
Several testing procedures were used to determine the various thermal properties of both treated and untreated fibers. For these tests, a Thermal Gravimetric Analyzer (TGA) was utilized to precisely measure the mass of fibers as they were subjected to various temperature profiles.

To determine a safe operating range, fibers were slowly heated from room temperature at a constant rate until the fibers completely broke down thermally. Hydrogen gas was pumped into the test chamber of the TGA to prevent combustion as well as control the humidity. To determine an optimum drying temperature, several candidate temperatures were chosen and fibers would be dried at one of the chosen constant temperature for several hours. To measure the effects of wetting and drying, a sample of fibers would be dried for 3 hours, then immediately be placed in a water bath for 30 mins. This same sample of fibers would then undergo a second drying session for 3 hours. The results of the two drying sessions would then be compared. A final measure to be explored thermally was idle time. Idle time refers to the amount of time treated fibers spent in storage in the temperature-humidity control chamber. It can be seen as a

measure of the shelf life of a treatment. Treated fibers with various idle times would undergo the same drying profile. The results would then be compared.

### **Cross-Sectional Area Determination**

In order to measure things such as tensile strength, the cross-sectional area of individual banana fibers will need to be determined. Banana fibers have a significant amount of voids within their volume, as shown in Figure 5. These voids make up 30%-60% of the total volume fraction of the fiber. While this is significant, the voids within the fibers are only around 10  $\mu\text{m}$  in diameter, which is too small for the LDPE matrix to enter into during processing [3]. Thus, the voids play little role in enhancing the overall strength of the composite material. As such, they will be included in the cross-sectional area of the fiber for strength calculations.



**Figure 5: SEM micrograph of the cross-section of a banana fiber**

### **Single Fiber Tensile Testing**

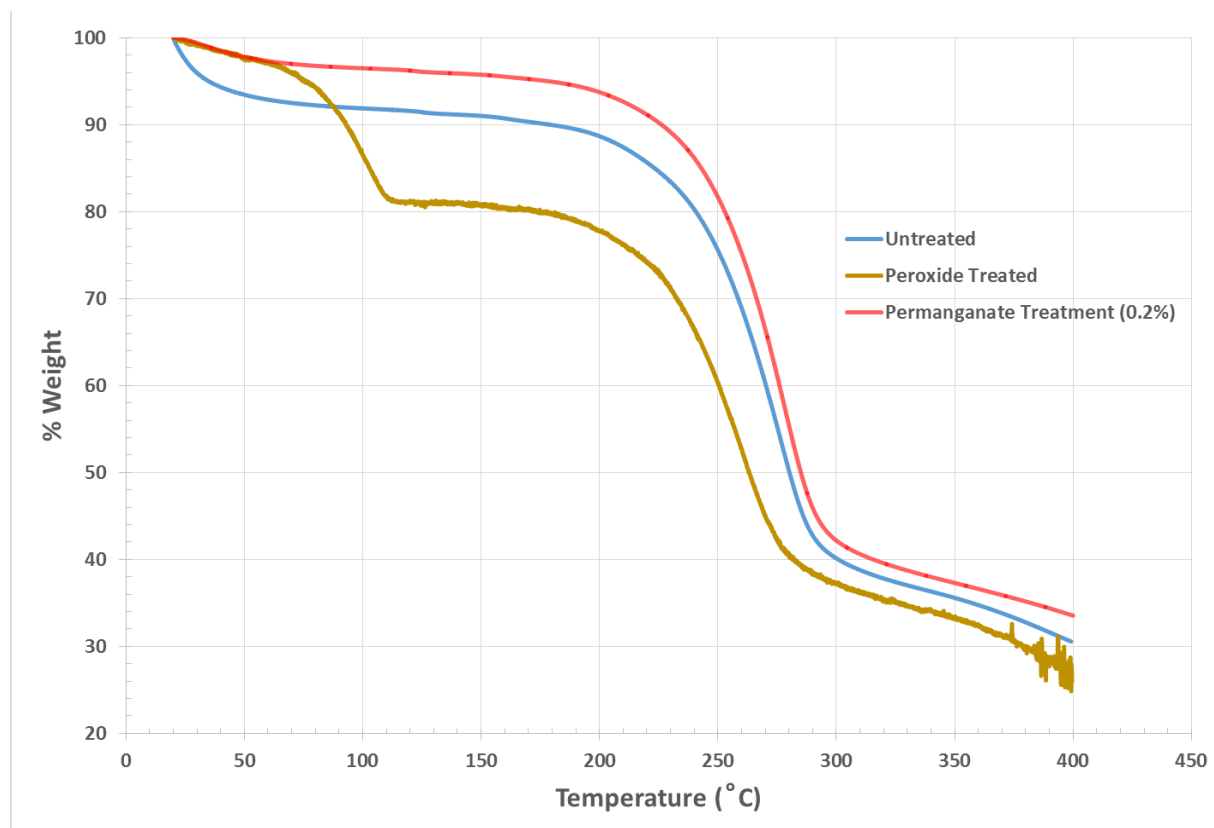
In order to test the effects that the chemical treatments have on the individual strength of the banana fibers, a Dynamic Mechanical Analyzer (DMA) testing machine was used. With a tensile range of 0-18 N, the DMA allows for precise measurements of the loads subjected to individual banana fibers. For both treated and untreated cases, individual fibers were subjected to a tensile load from a constant rate of extension (0.5 mm/min) until fracture. The effects of idle time were also explored. Idle time refers to the amount of time treated fibers spent in storage in the temperature-humidity control chamber. Fibers with the same chemical treatment but different idle times were subjected to the same tensile test, and the results were compared. The cross-sectional area of a sampling of each type of treated fiber was then determined using SEM imaging and the areas were averaged for each type.

### *Results and Discussion*

#### Thermal Treatment

Results from the fiber burnoff test for both untreated and treated fibers are shown below in Figure 6.

As can be seen, all cases first display a relatively small dip in weight. This can be associated with the initial moisture in all of the fibers because the weight stabilizes shortly after 100°C, or the boiling point of water. Shortly after 200°C, all cases suddenly begin to drop drastically in weight. Due to the magnitude of this drop, it can be safely assumed that it is due to thermal degradation. Therefore, it can be concluded that a safe operating temperature range for banana pseudo-stem fibers is less than 200°C. Many types of plastics, including LDPE, are typically processed at temperatures lower than this, making banana pseudo-stem a viable choice for natural reinforcement.

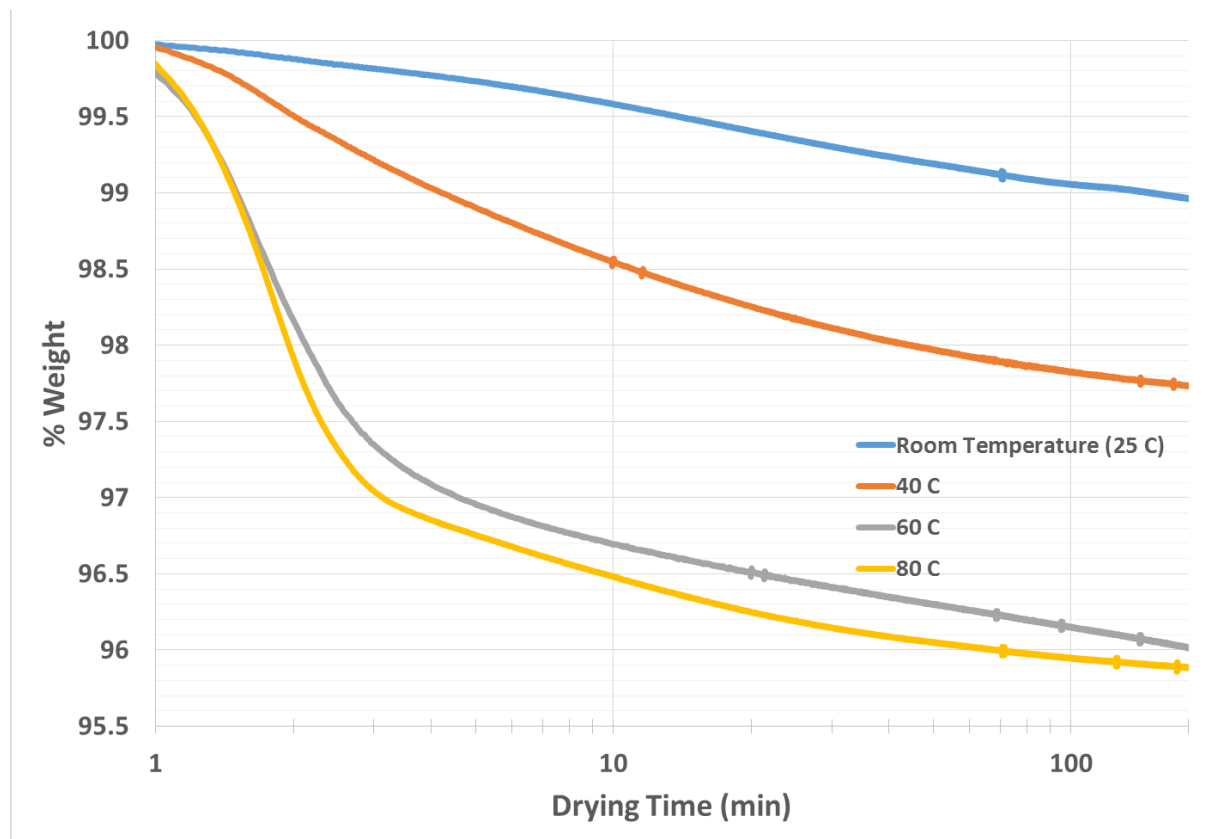


**Figure 6: Burnoff Test for Banana Pseudo-stem Fibers**

Figure 7 compares the results of different candidate drying temperatures for untreated fibers. The candidate drying temperatures are 40°C, 60°C, and 80°C. 80°C was chosen as the maximum candidate temperature because the softening temperature of LDPE is around 100°C.

When comparing the different drying temperatures, it becomes apparent that there is very little difference in the effectiveness of 60°C and 80°C. Both are more effective than 40°C, and though 80°C is slightly more effective, 60 °C is farther away from the softening temperature of LDPE, and thus would be more effective for drying out fibers without affecting the composite material as a whole.

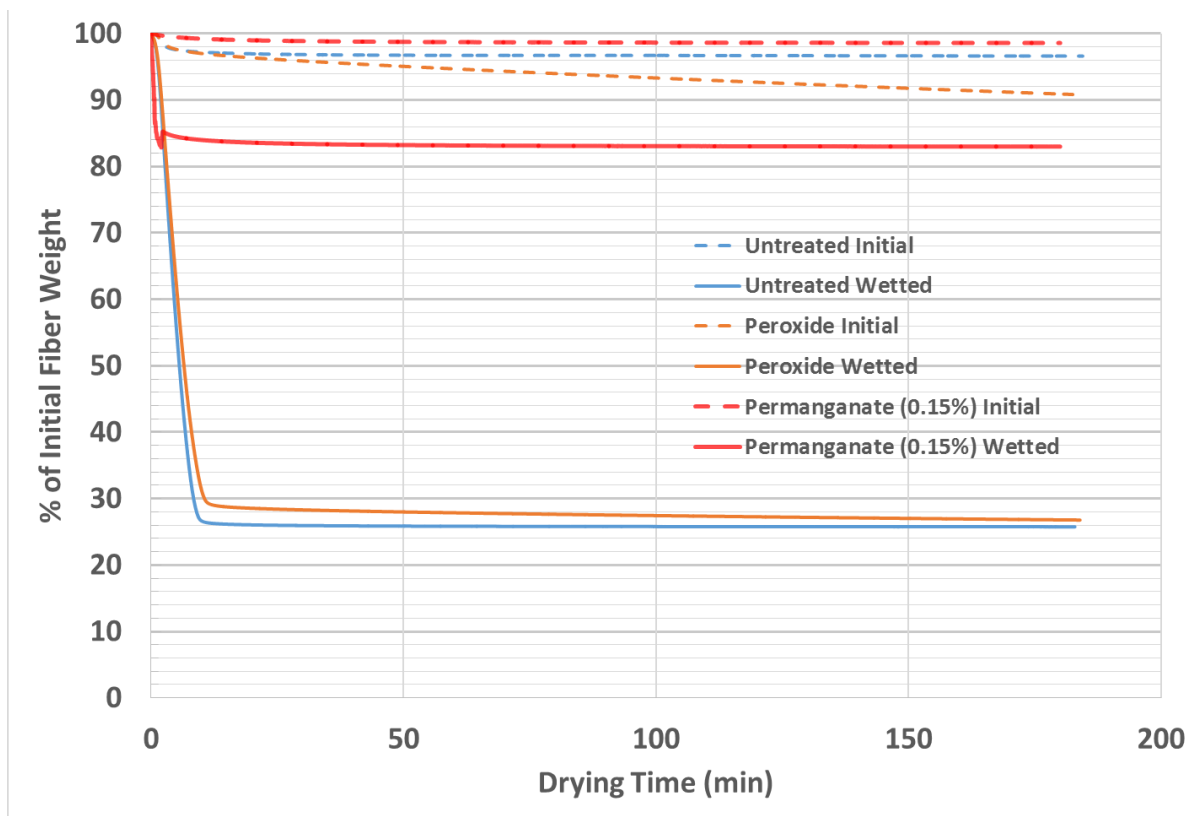




**Figure 7: Iso-Temperature Profiles for Untreated Banana Fibers**

With a drying temperature of 60°C selected, treated and untreated fibers were subjected to a round of wetting and drying tests. The results from these tests are shown below in Figure 8.

As can be seen, In the initial pre-wetted stage, both the treated and untreated fibers have very similar drying profiles. After being wetted, however, the peroxide treated fibers mimic the drying profile of the untreated fibers very closely, but the permanganate treated fibers do not. This suggests that the peroxide treated fibers do not do much to decrease the hydrophilicity of banana fibers, because they absorbed just as much water as the untreated fibers did. The permanganate treated fibers appear to absorb less water, as less weight was expelled from the fibers after being wetted. This would suggest that the permanganate treatment does decrease the hydrophilicity of banana fibers.

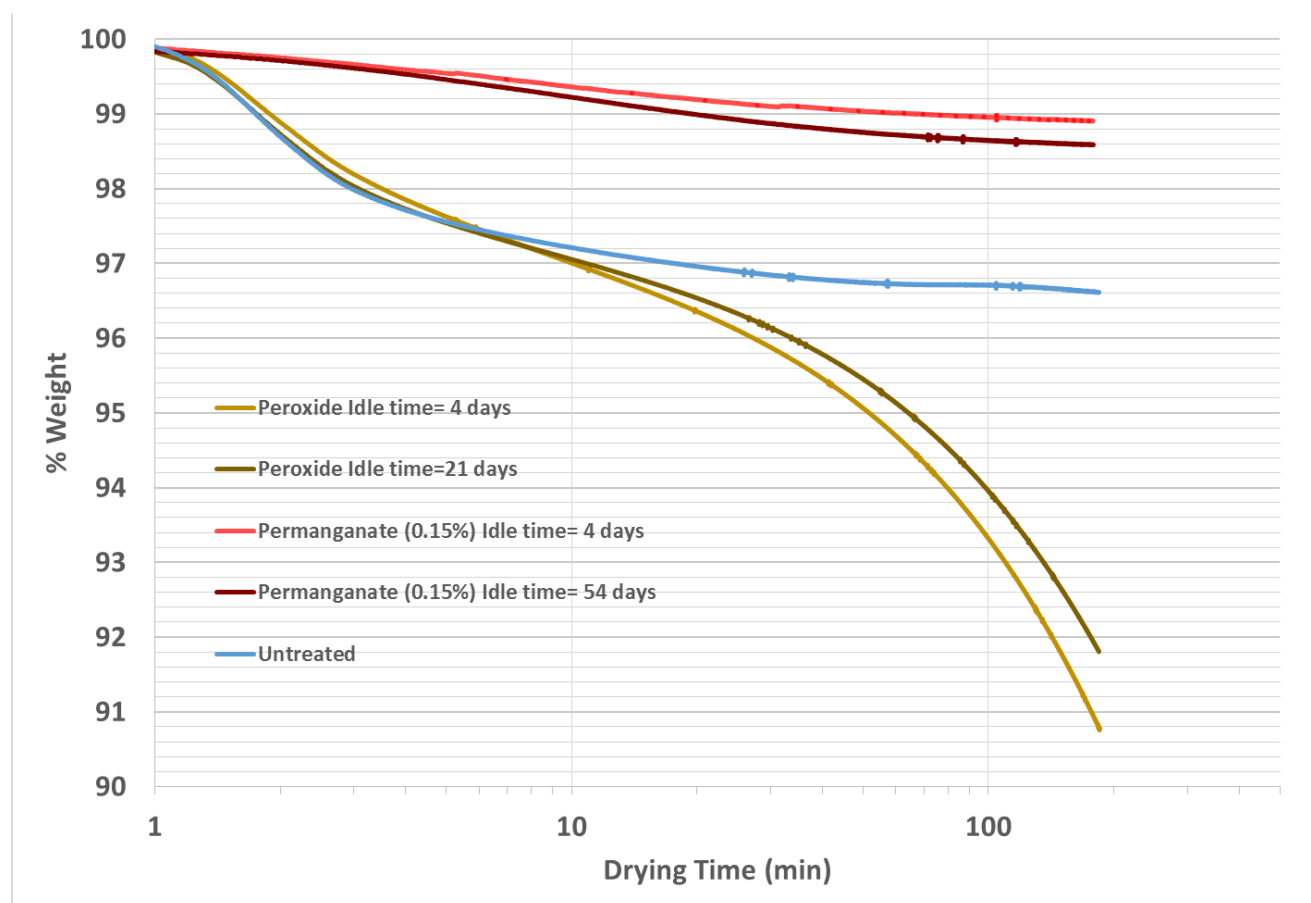


**Figure 8: Drying Profiles for Wetted Banana Fibers, 60 C**

Finally, the effects of idle time on the treated fibers were explored by comparing the results of drying tests from batches of fibers with different idle times. Idle time refers to the amount of time treated fibers spent in storage in the temperature-humidity control chamber. The results of these tests are compared in Figure 9.

As idle time increases for both treatments, it does not have a significant effect on the overall drying profile, suggesting that the thermal properties as a whole do not significantly change over time. Though the change is small, both treated cases begin to more closely mimic the drying profile of the untreated case as idle time increases. As mentioned before, the permanganate treatment suppresses the hydrophilicity of the fibers, and thus there is less water weight for them to expel. The rationale behind the peroxide treatment behavior is less certain. It

can be theorized that the peroxide treatment leaves a protective chemical layer on the fiber surface that is slowly evaporating or disintegrating at elevated temperatures.



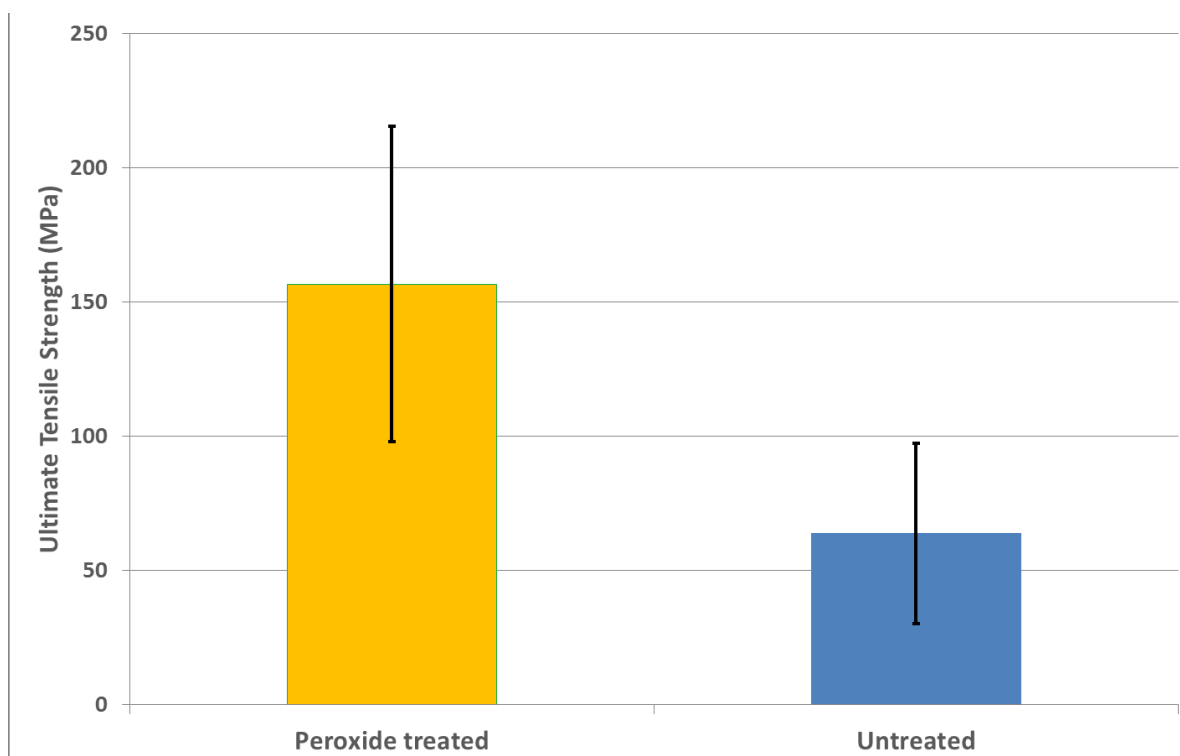
**Figure 9: Drying Profiles for Treated Banana Fibers, Unwetted, 60 C**

### Single Fiber Tensile Test

Results from the peroxide treated single fiber tensile test is shown below in figure 10. The untreated case is included for comparison purposes.

As can be seen, the peroxide treated fibers seem to have a higher ultimate tensile strength than the untreated case. One possible reason for this is that peroxide treatment doesn't degrade the surface of the fibers. This would explain why its strength is not less than the untreated strength. As to why it is stronger, it could be that the peroxide treatment forms a

protective layer on the outside edge of the fibers, making them more durable than the untreated case.



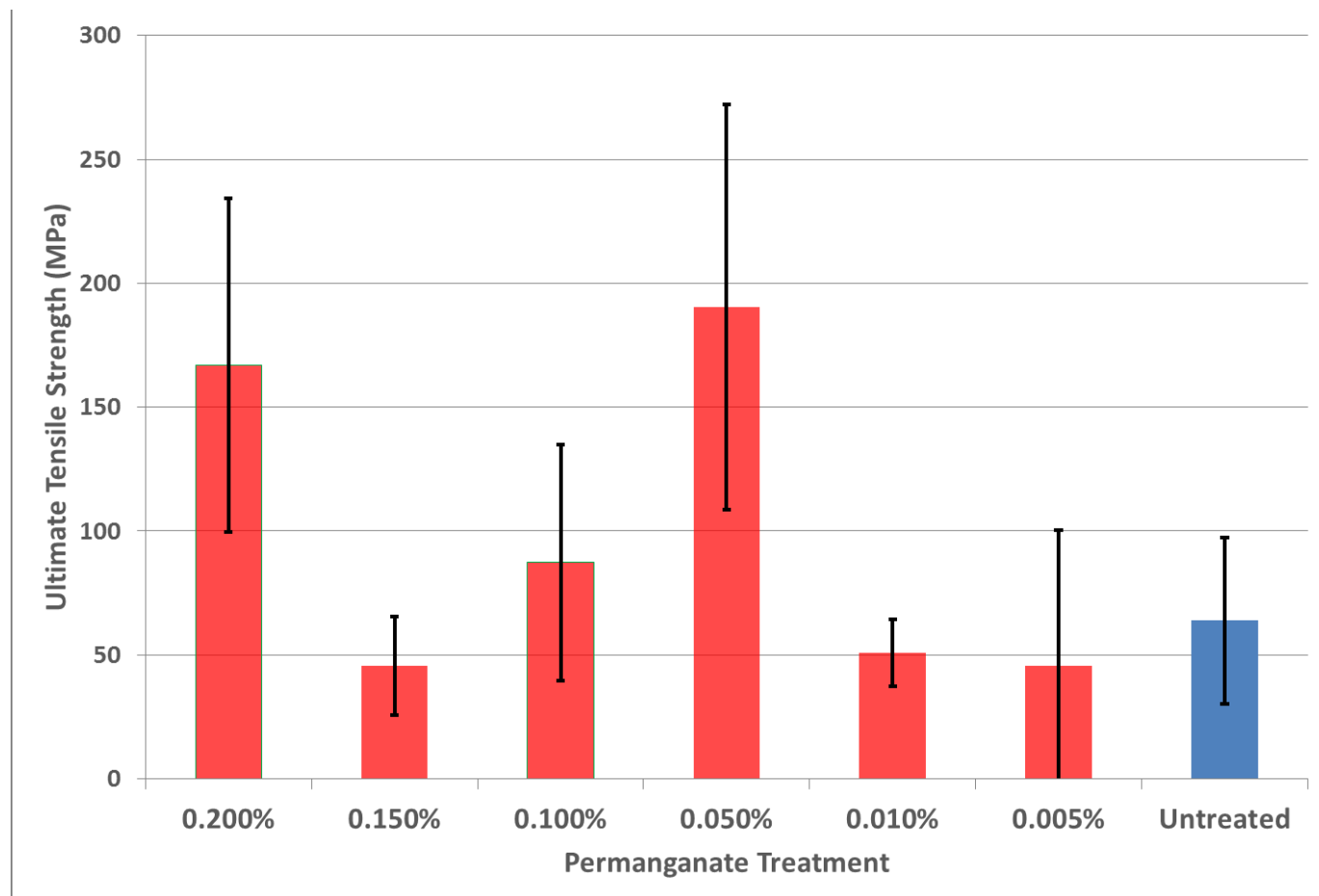
**Figure 10: Ultimate Tensile Strength (Median), Peroxide Treatment**

In similar fashion to Figure 10, Figure 11 presents the results for the permanganate treated single fiber tensile tests.

On first glance, the results seem to be a little confusing, as there appears to be little correlation between the concentration of permanganate treatment and the ultimate tensile strength. There is exceptionally wide variation with the data in almost every case. It was expected that the data would be a lot closer to the results represented in the 0.005%, 0.01%, and 0.15% permanganate treatments, as permanganate treatment does lead to some degradation of the surface of the fibers.

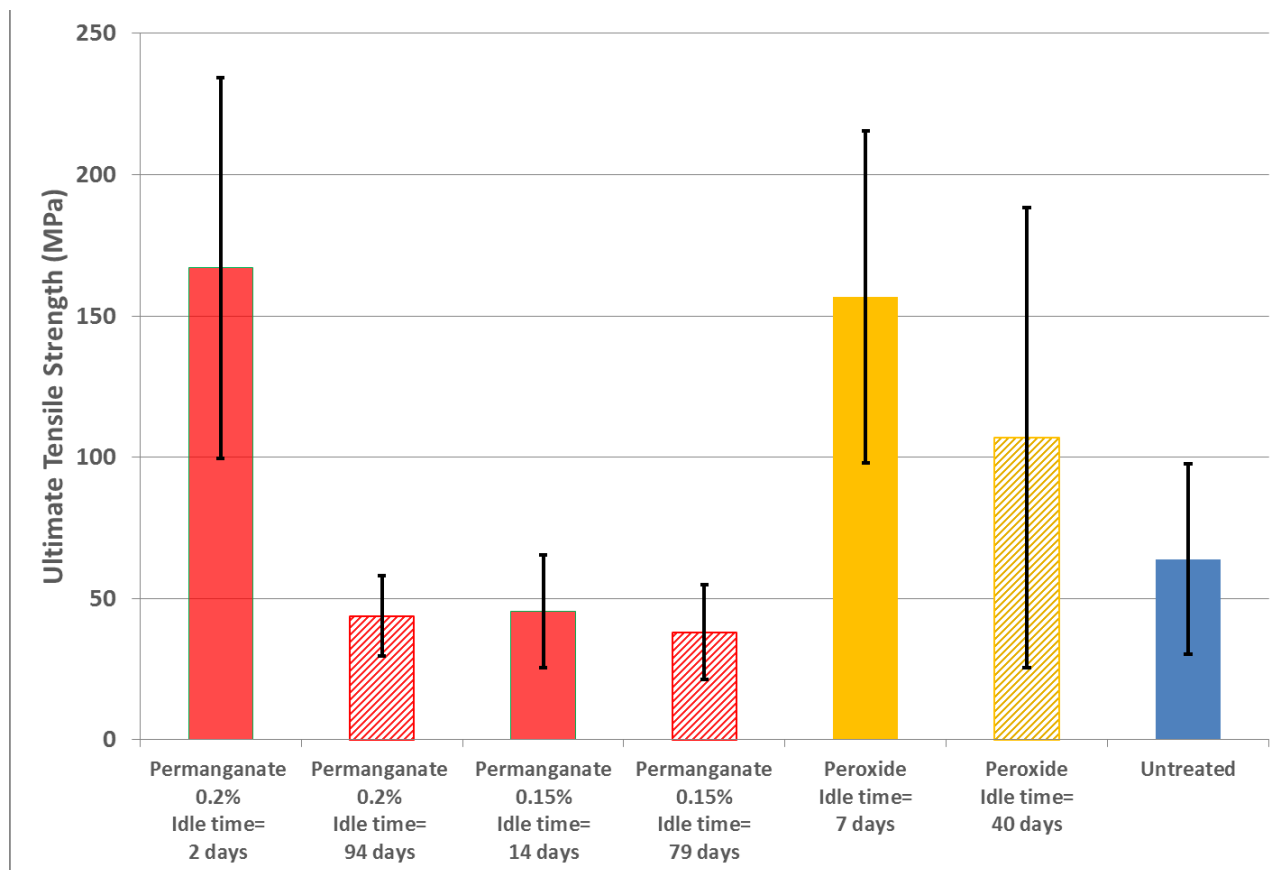
A possible explanation for these results was formulated upon closer examination of the test fibers' cross-sectional area in the SEM. Though due diligence was used in the selection and

preparation of single fiber tensile samples, it is possible that some “single fibers” were actually a combination of a few exceptionally small fibers. Because the permanganate treatment roughens the surface and promotes cross-linking, it is possible that the perceived extra strength of the fibers is actually the result of the increased friction of multiple fibers sliding past each other.



**Figure 11: Ultimate Tensile Strength (Median), Permanganate Treatment**

Just like in the thermal tests, the effects of idle time on the tensile strength were explored by comparing the results of tensile tests from batches of fibers with different idle times. Idle time refers to the amount of time treated fibers spent in storage in the temperature-humidity control chamber. The results of these tests are compared in Figure 12:



**Figure 12: Ultimate Tensile Strength (Median), Idle Time Comparison**

For all treated cases, the ultimate tensile strength does decrease with increasing idle time. For permanganate treatments, the severity of this drop appears to change drastically with the concentration: the 0.2% treatment drops to less than 1/3 of its supposed initial strength, whereas the 0.15% treatment hardly drops at all. While the source of the severity is unclear, the overall trend is more explainable. The permanganate treatment is a relatively caustic reaction, thus it is possible that the remnants of the treatment after drying continue to slowly degrade the fiber surface. For peroxide treatment, the drop is more moderate, losing around 1/2 of the extra strength it had over the untreated case over the course of 30 days. A possible explanation for this is tied to the previous theory of the protective chemical layer on the fiber's surface. If it is

evaporating or disintegrating over time, the fiber would likely lose some of the extra strength associated with it and more closely mimic the untreated strength.

### *Conclusion*

In dealing with the bonding issues of banana fibers and LDPE, peroxide and permanganate treatment are both possible solutions that chemically treat the banana fibers to decrease hydrophilicity and/or enhance the bonding strength of the fibers. These treatments appear to not have a detrimental effect on the thermal properties of the fibers. A safe operating temperature range for banana pseudo-stem fibers is 200°C or less. Permanganate treatment seems to be more effective at curtailing the hydrophilicity of the fibers than peroxide treatment. These properties do not significantly change with idle time.

Peroxide treatment appears to enhance the overall strength of banana fibers. It is theorized that this may be due to a thin protective film that forms on the surface of the fibers due to the treatment. The effects of permanganate treatment do not appear to be correlated with its concentration. Some concentrations reside at around the same value below the untreated fiber strength, while other concentrations shoot wildly high in strength above the untreated case. This may be due to the fact that some single fibers were found to be multiple tiny fibers, and its strength was enhanced due to the increased friction between fibers. In all cases, strength decreased with increasing idle time. The severity of this decrease varied based on treatment and concentration.

### *Future Work*

The next steps in this research involve looking at how these treatments affect the interfacial shear strength of the fibers. Once quantified, the overall properties of the composite material will be measured with peroxide and permanganate treatment. The processing involved in making the composite will be carefully considered to minimize the swelling described by

Weed [3]. The composite material will undergo standard tensile testing, and the impact properties of the material will also be explored.



## Works Cited

[1] Li, Xue, Lope G. Tabil, and Satyanarayan Panigrahi. "Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review." *Journal of Polymers and the Environment* 15, no. 1 (January 1, 2007): 25–33. doi:10.1007/s10924-006-0042-3.

[2] Burdick, D., "Banana Heart Recipes," Environmental Challenges of the Philippines

[Online]. Available:

<http://ecop.pbworks.com/w/page/18520479/Banana%20heart%20recipes%200809>.

[Accessed: 09-Dec-2013].

[3] Weed, Joshua. "Investigation of Interfacial Bonding in Banana Fiber-Reinforced Composites: Ascribing Global Economic Value to Unique Renewable Resources in Developing Countries."

(2014)

[4] Joseph, Kuruvilla, Sabu Thomas, and C. Pavithran. 1996. "Effect of Chemical Treatment on the Tensile Properties of Short Sisal Fibre-Reinforced Polyethylene Composites." *Polymer* 37 (23): 5139–49. doi:10.1016/0032-3861(96)00144-9.