DETERMINING AN INORGANIC MINERALIZATION PROCESS TO INHIBIT ORGANIC DEGRADATION AND PRESERVE THE DIMENSIONAL STABILITY OF BAMBOO

California Polytechnic State University

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Project Title: Determining an Inorganic Mineralization Process to Inhibit Organic Degradation and Preserve the Dimensional Stability of Bamboo

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Materials Engineering Department

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Abstract

Bamboo is a renewable natural composite with potential as both a construction and synthesis material. The components of bamboo are cellulosic vascular bundles imbedded within a lignin matrix that create a porous hierarchical structure. This structure has low density while maintaining high strength, toughness, and elasticity, but is susceptible to organic decay. A study was performed to evaluate the effect of varying bamboo culm age (1-3 years) and density (0.72-1.05 g/cm^3) on the ability to control inorganic mineral precipitation to prevent degradation and preserve dimensional stability of the organic matrix. Internodal culm samples were immersed in solutions of sodium silicate, citric acid, and mineral water (NaSi:TCA:H_2O) in volumetric ratios. Immersion periods varied from 30 seconds (evaporative precipitation) to 24 hours (acid-catalyzed soak diffusion). Water adsorption isotherms were modeled using Fick's second law of diffusion and adsorption coefficients indicate that an evaporative precipitation treatment reduced the absorption coefficient of bamboo by 10%. Soak diffusion treatments increased the absorption coefficient and these results are representative of significant lignin leaching during processing and correlate to a reduction in total bamboo hydrophobicity negating the effects of the mineralization treatment. SEM analysis showed culms exposed to solutions of pH 6.5-7.3 showed limited mineralization, pH 8 evidenced mineral precipitations primarily within cell wells, and pH > 8.5 showed significant cellular decomposition and mineralization within pores. XRD analysis was performed that indicated the preservation of crystalline cellulose in evaporative precipitation treatments as well as peaks that correspond to sodium-rich feldspars and silica.

1.0 Introduction

1.1 Broader Impacts

It is critical that we understand how to design processes, systems, and components that function to meet design needs within the context of the broader impacts on society. Realistic constraints such as sustainability and manufacturability address how bamboo and a protective treatment recognize and address natural design limitations.

1.1.1 Sustainability

Sustainability encompasses the responsible management of resource use that includes how environmental, economic, and social aspects remain productive and balanced. The construction industry has been identified as the primary consumer of both energy and materials in most countries [1]. The need to investigate sustainable construction practices has allowed bamboo to be realized as a locally grown replacement for steel reinforcement in concrete [1,2]. Attainable technical information regarding the potential of bamboo is critical because the lack of reliable technical information often forces consumers to ignore local materials in favor of expensive materials for which the information is freely available, such as Portland cement and steel. Furthermore, in many countries building codes are not established or corners are cut due to the high cost and unavailability of products such as steel [1,2]. Using bamboo allows local engineers and construction managers the ability to improve the quality of their products without negative economic or environmental interference. Bamboo has one fiftieth of the embodied energy of steel and is 35% more carbon negative than an equivalent density of trees as shown in Figures 1 and 2.

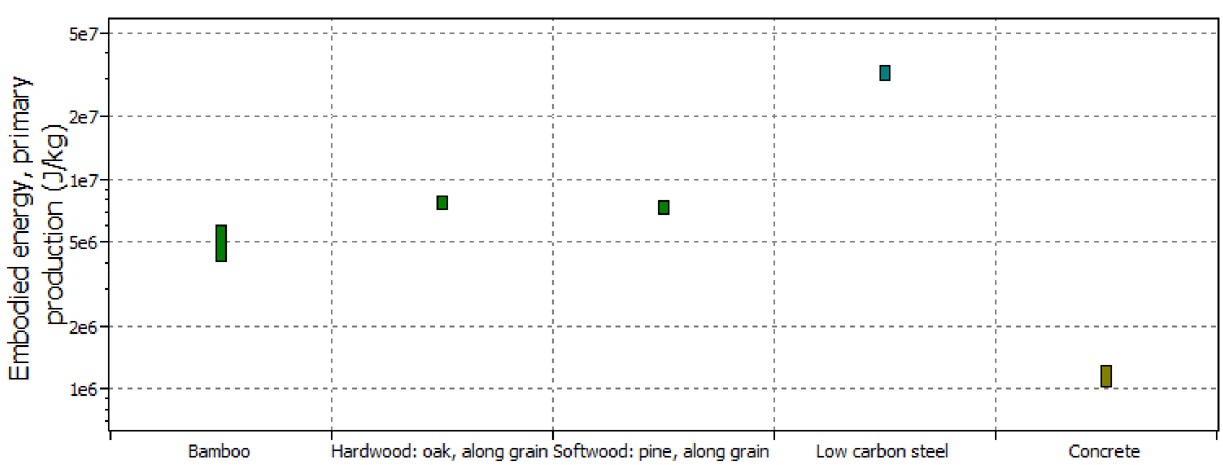

Figure 1. Embodied Energy of different natural materials as they compare to steel and concrete.

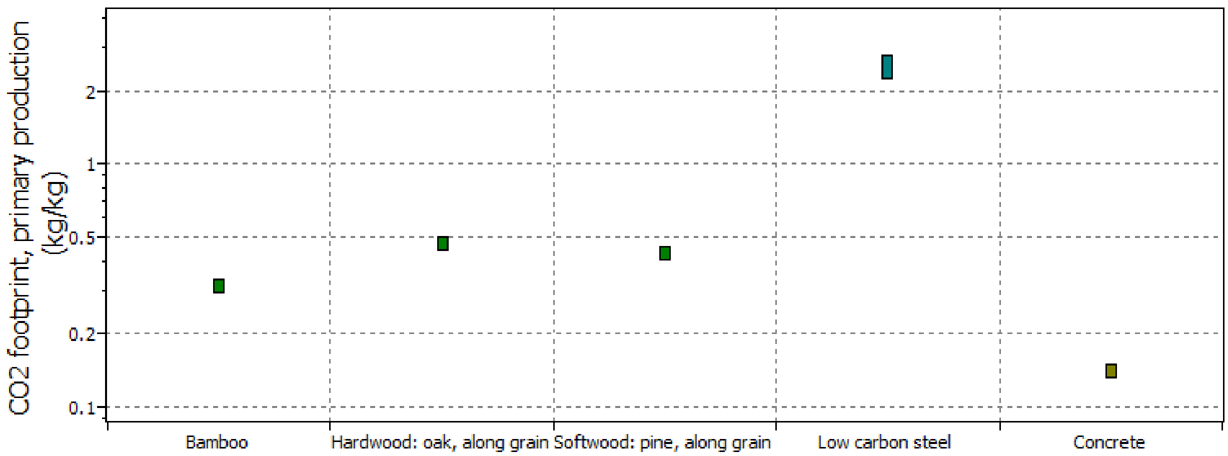

Figure 2. CO2 Production of different natural materials as they compare to steel and concrete.

Local annual production of bamboo also yields regular local employment due to annual harvesting and is resistant to erosion and deforestation that is associated with harvesting trees over longer durations [3]. Both of these concepts are represented by Figure 3 below.

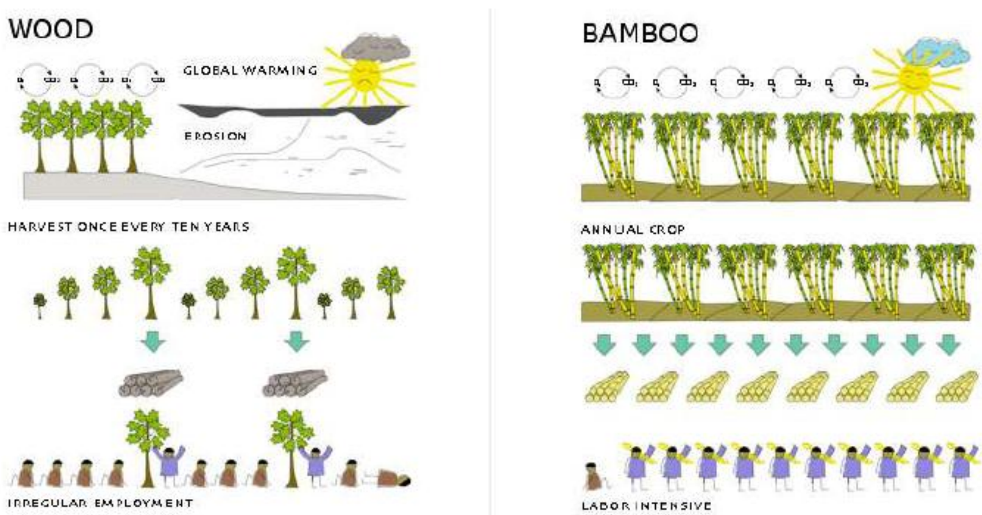

Figure 3. Correlation between growing cycle, regular employment, and erosion for wood and bamboo.

1.1.2 Manufacturability

My project specifically focuses on how bamboo can be processed and treated to preserve its strength without the use of toxic non-local chemicals. This deals with the indirect longevity of built structures that is a result of manufacturability. Manufacturability describes the process of taking a raw material and converting it to a finished product. This aligns with my projects aim to develop a sustainable process to locally harvest bamboo as raw material and convert it into a structural material. Current bamboo

curing and preservation involves the absorption of toxic chemicals that often leak from the bamboo over time [3]. In avoiding the toxicity of these treatments, bamboo becomes significantly more desirable material in addressing sustainable structural materials due to the low environmental impact, low toxicity, low economic cost, and high performance [1,2].

1.2 Background

Nearly all biological materials are high performing composites that are constructed from a bottom's up design approach. Bamboo is an example of a natural structural material largely in use in its raw form, but protective treatment information is either not well distributed or well known. It is important throughout this study that bamboo is compared and contrasted with wood in order to take advantage of a more comprehensive literature background regarding wood. In this way, an understanding of wood allows for a better understanding of bamboo's limitations, benefits, and unknowns. One primary difference is that bamboo is defined as a giant grass of the family bambusoidae and thus many ASTM standards and specifications are ill-suited when bamboo is analyzed as a true wood.

In order to prevent degradation and preserve the dimensional stability of bamboo this study emphasizes a biomimetic approach and looks toward a natural process. Petrification is a mineralization process that creates a mineral psuedomorph of a natural structure, preserving its dimensions exactly. Petrification is often referred to as permineralization and occurs primarily with either silica or calcium. Silica was chosen for my study based on the abundance of silica based source materials and a more thorough literature review. Knowledge of how a mineralization based treatment provides a barrier for water adsorption is a critical step in determining the effectiveness once implemented as a structural reinforcement. It also identifies a recommended extension of this study to analyze how a mineralization treatment of bamboo bonds to concrete.

1.2.1 Properties and Structure of Bamboo

Bamboo has a high level of anisotropy and can be analyzed as a long fiber reinforced composite [4,5]. Bamboo's superior properties are derived from its complex, porous, hierarchical structure. It has high tensile strength to specific weight and elastic modulus to specific weight values that are approximately 6 times greater than steel as shown in Figure 4 [6].

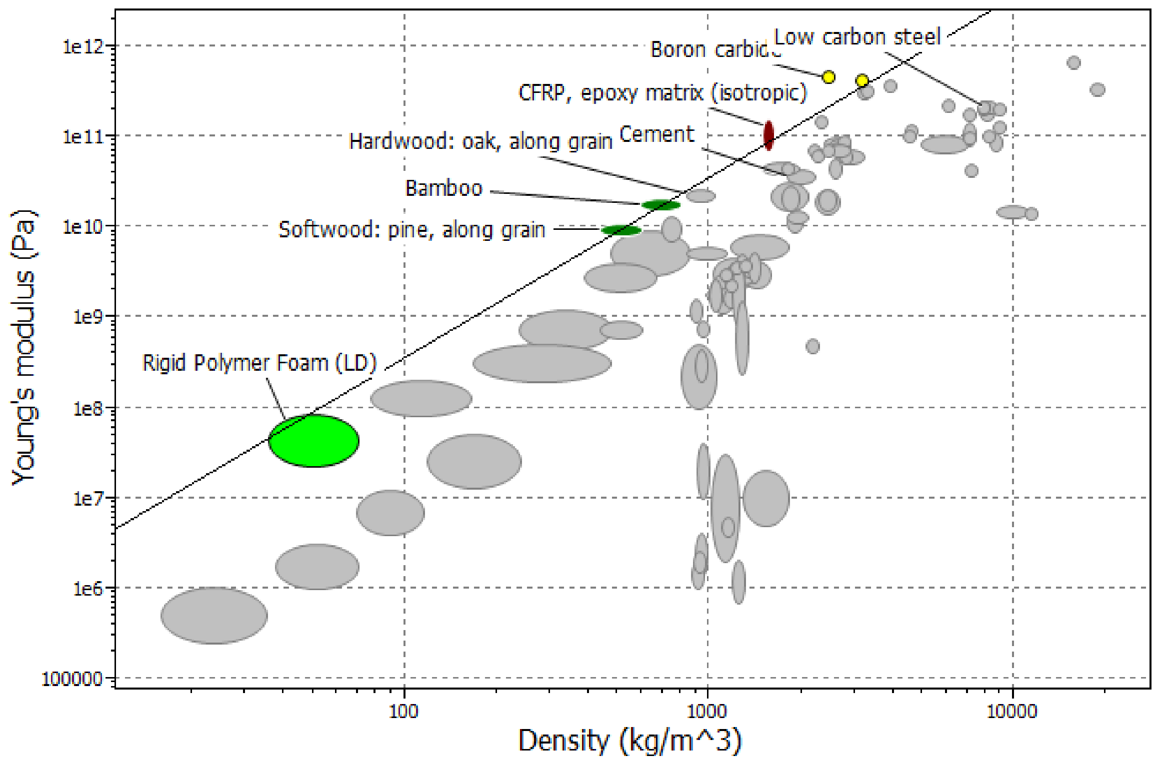

Figure 4. Cambridge Engineering Selector Plot of $E^{1/2}/\rho$. Materials with ratios below the line have properties less than bamboo and materials above the line have greater ratios than bamboo. The enclosed area for each material represents variable material properties [7].

The most pronounced feature of bamboo on the macro scale is the prominence of nodal and internodal sections as seen in Figure 5. The above ground portion of bamboo is referred to as the culm, consisting of hollow cylindrical shells, called internodes, divided by transverse diaphragms, called nodes. The hallow cavity is referred to as the pith and thus the innermost part of the culm wall is called the pith ring and consists of thick, heavily lignified parenchyma cells. The outermost part of the culm wall is called the cortex and consists of an epidermis and cutinized waxy layer. It is well known that the cortex and pith ring are hydrophobic, but limited knowledge is available regarding how these layers are formed and how well they repel water. Figure 5 shows an initial study of bamboo performed to understand the differences between the contact angle measured at the waxy epidermis, a cellulose bundle, and the lignified pith.

Also represented in Figure 5 is the internal microstructure of bamboo and the prominence of cellulose vascular bundles embedded within the ligneous matrix. Fiber density varies along the thickness and

height. Fiber density increases towards the exterior epidermis due to a gradient of stress distribution when growing [1]. The vascular bundles consist of metaxylem vessels and fibers; vascular bundles contribute to the elastic modulus of the bamboo while the fibers contribute to the tensile strength [8].

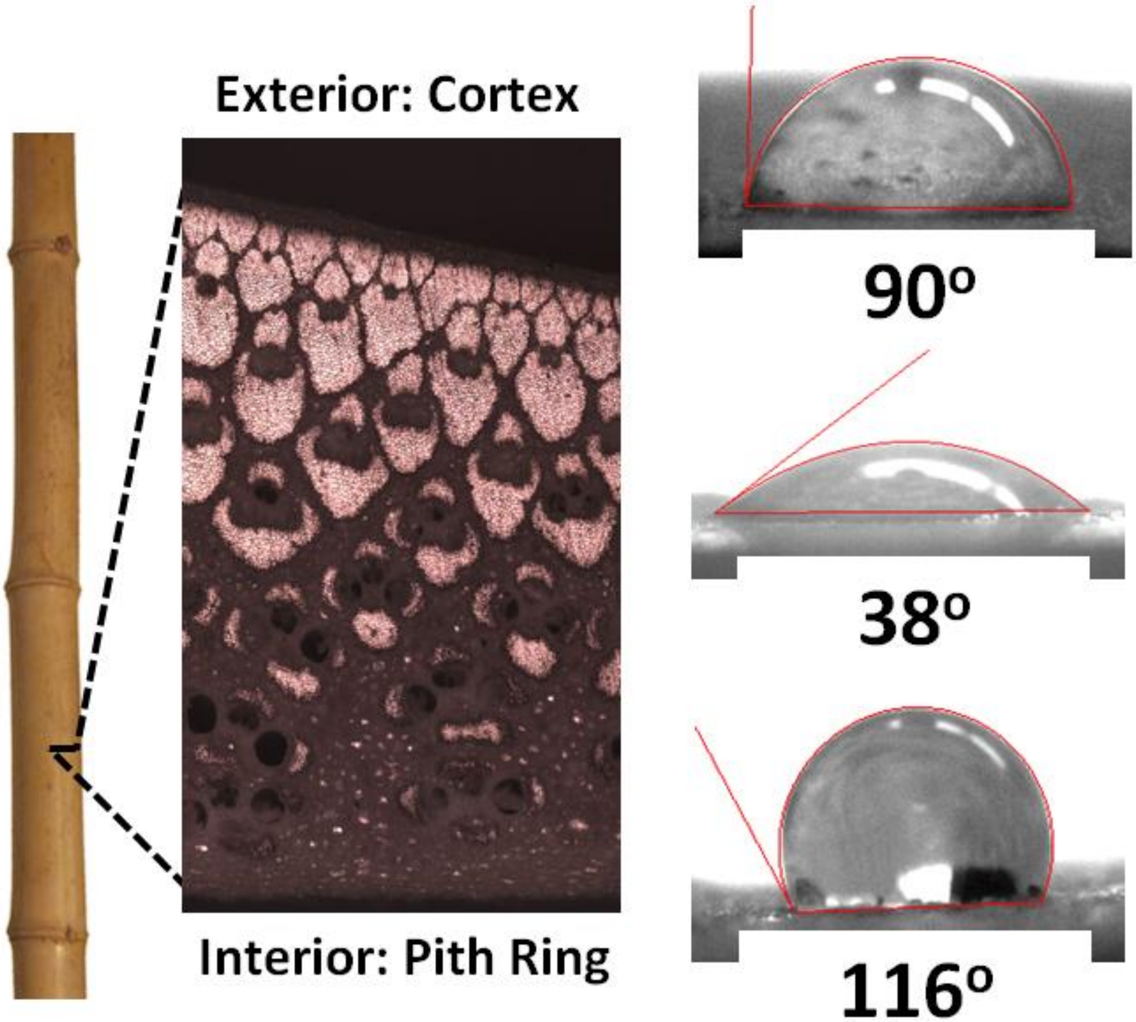

Figure 5. Cross-section of a bamboo culm internode illustrating increasing fiber density and decreasing size towards the exterior cortex. Representative contact angles for water are also shown correlating to their location on the microstructure.

The middle of the above microstructure is referred to as the culm wall and consists of approximately 50-52% parenchyma cells, 8-10% conducting vessels, and 40% fibers. These relative amounts of cells give us two important pieces of information: bamboo should be analyzed as a composite and thus the ratio of matrix (parenchyma cells) to reinforcement (fiber bundles) is 1:1; secondly, it is significant that bamboo contains about 10% water conducting tissues compared to 20-30% in hardwoods and 70% in softwoods

[8]. This lower volume of conducting metaxylem vessels is representative of the absence of ray cells and further determines the need for axial rather than radial protection.

1.2.2 Chemical Structure of Bamboo

In section 1.2.1 the idea of water adsorption was addressed through the comparison of the contact angle at either cellulosic or lignified areas. Cellulose is a polymer formed by the condensation of D-glucose monomers bonded through glycosidic bonds. Terminating hydroxyl groups contribute to substantial hydrogen bonds and greatly increase the hydrophilic nature of cellulose as seen in Figure 6.

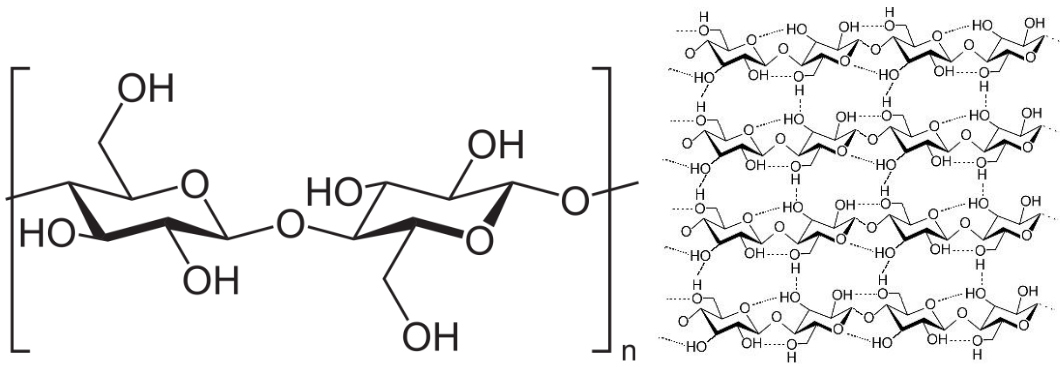

Figure 6. (Left) D-glucose monomer showing terminating hydroxyl groups, (Right) Cellulose Polymer Structure showing hydrogen bonding.

Lignin on the other hand is a much more complex polymer responsible for the crosslinking between different cellulose components of the culm wall. This crosslinking is shown in Figure 7 and contains a diversity of functional groups including ethers, esters, and alkanes that yield a high degree of hydrophobicity.

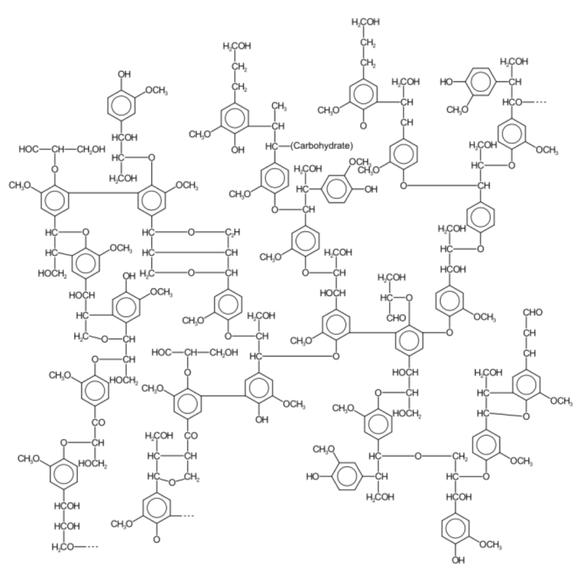

Figure 7. Chemical structure of lignin showing the large size and significant cross-linking of the molecule.

1.2.3 Natural Degradation of Bamboo/Wood

The lifetime of bamboo without treatment is limited to 5 years [8]. One primary reason for the shorter lifetime compared to other materials is the high variety of degradation modes. These environmental degradation modes can be dived into two broad categories, biological and abiotic, both of which relate to the presence or fluctuation of excess water in the system. Biological refers to bamboos susceptibility to attack by fungus or insects. Fungal attack consists of surface mold, stain fungi, and decay fungi while insect attack is caused by termites or boring beetles. Abiotic refers primarily to weathering or pyrolysis and is caused by rapid drying, rapid swelling due to water absorption, temperature fluctuations, degradation of biopolymers in UV radiation, dirt/sand abrasion, or addition of excess heat.

Another impediment to the utilization of bamboo in a concrete system is the failure mechanism that results from the swelling of bamboo during the curing of concrete.

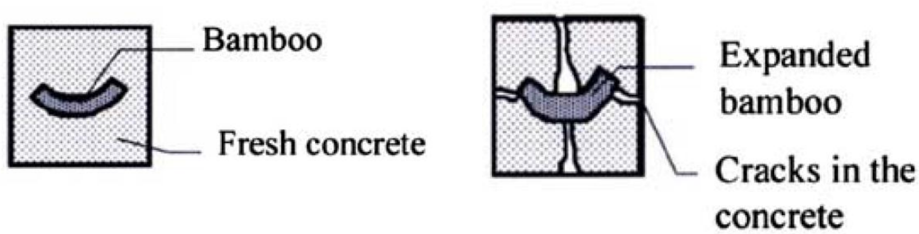

Figure 8. Swelling of bamboo during concrete curing causing cracks and premature failure.

In order to address these problems, current chemical treatments use preservatives that are either non-fixing, toxic, or require intricate processing procedures. Here a need has been identified for a non-toxic fixing solution that is easily applied and reduces bamboo's sensitivity to water.

2.0 Experimental Procedure

It is important to remember that the goal of this experiment is to create a silica rich solution with the ability to penetrate bamboo and coat the cellulose fibrils without significant lignin leaching. Silica must be either in solution or existing as a small colloid to penetrate the bamboo. The reason for the small particle size was to increase the surface area, increasing solubility, and to allow the silica and other mineral cations to diffuse through micropores (8-36 Å) in the bamboo cell wall.

2.1 Bamboo Samples

Bamboo culms of two varieties and two age ranges were donated by Paso Bamboo Farm and Nursery located in San Luis Obispo county, CA: Madake Gaint Timber, Phyllostachys bambusoides, 1 year and 3 years old; and Henon, Phyllostachys nigra, 1 and 2.5 years old. Both varieties are of the giant timber family used for their strength, size, and high yield. Madake timber has large, straight, thick walled culms with larger internodal areas whereas Henon culms are white and gray with a thinner culm and rough epidermis.

Bamboo culm samples were cut into approximately 10cm sections of internodes. Multiple inner and outer diameters were measured to model bamboo as a slight ellipse rather than a circle to give more accurate surface areas, thicknesses, and densities.

Figure 9. Internodal sections of 1 year old Madake timber showing relative sample size and inherent variation of samples.

2.2 Absorption Coefficients

In order to determine absorption coefficients of water into different varieties/ages/treatments of bamboo, 5 internodal sections of each permutation were immersed in deionized water for 3 hours. In addition to initial mass, weight measurements of each sample were taken every 10 minutes by removing the culms from solution and using a damp cloth to wipe excess water from the sample before weighing.

2.3 Moisture Content

In order to determine the moisture content of different varieties/ages of bamboo, 3 internodal sections of each permutation were weighed and then inserted into a furnace at 102 ^{o}C for 12 hours. It was important that the temperature be over 100 ^{o}C because this presents any fungus from growing on the bamboo while in the furnace. Weight measurements were taken after 12 hours and the samples placed

back into the furnace at 102 ^{o}C in one hour increments until the weight fluctuation between each measurement was less than 2%. Moisture content represents the relative amount of water weight compared to the weight of the dry bamboo.

2.4 Preservative Treatment

2.4.1 Solution Application

Treatment methods are based on modified treatments currently used with toxic fixing chemicals and were chosen for ease of application. The first method selected was soak diffusion which consists of bamboo samples being immersed in solution and sunken by weights for 24 hours such that preservative moves into the bamboo by concentration gradient while any mobile sap moves out due to osmotic pressure. The second method chosen was evaporative precipitation and consists of a 30 second "dip" into solution and then removal for the samples to air dry and cause mineralization as water evaporates.

2.4.2 Solution Chemistry

Internodal culm samples were immersed in solutions of sodium silicate, mineral water, and citric acid (NaSi:M-H_2O:TCA) by volumetric ratios as shown in Table I.

Table I – Representative Volumetric Ratios of sodium silicate, citric acid, and mineral water and their pH

	NaSi	M-H20	TCA
pH 7.2	1.00	120.00	5.00
pH 8.0	1.00	120.00	0.50
pH 9.3	1.00	80.00	0.50

These pH values were selected based on the solubility of silica and simple experiment regarding the ability of bamboo to retain integrity at varying pH as represented by Figure 10.

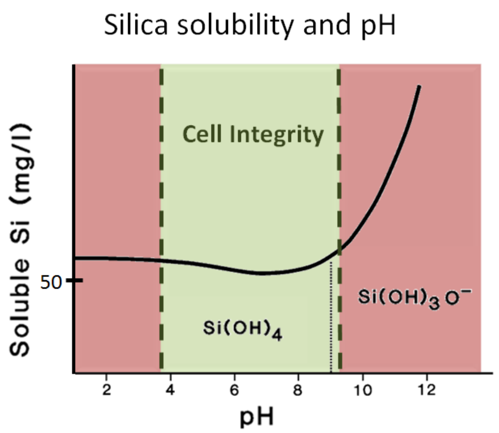

Figure 10. Silica solubility vs pH taking into account the integrity of bamboo cells.

Sodium silicate grade N was purchased from the National Silicates Company. Grade N specifies between 28-30% SiO_2 and a weight ratio of 3.22 SiO_2/Na_2O yielding a solution of between 37-38% solids. Mineral water was prepared by mixing fine montmorillonite clay with water and letting the solution sit for one week according to the solubility of silica with time. This process of solutionizing and settling can be seen in Figure 11.

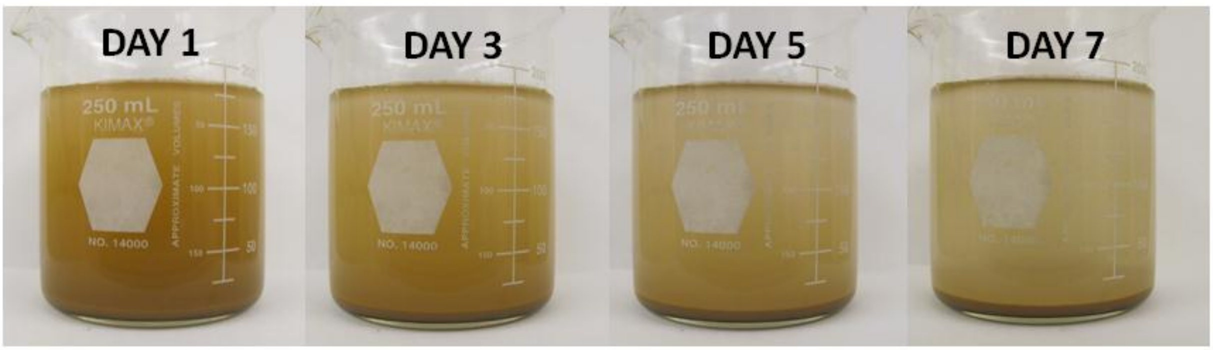

Figure 11. Montmorillonite Clay and water deflocculating and increasing solutionization of silica over the course of one week. Day 7 was poured off to be used as mineral water.

Approximately 25g of clay was used for 0.25 L of solution. The mineral water was then poured off into another container leaving solid clay behind. Before mixing, a mortar and pestle was used to reduce the particle size of a Montmorillonite clay mix purchased from Moltan Company. The mix contained 87-90% calcined montmorillonite clay, 7-10% crystalline silica (quartz), and <3% crystalline silica (crystobalite). It was important during grinding that a face mask was worn at all times to protect from inhalation of airborne crystalline silica. The chemical formula for Montmorillonite clay $Ca_{0.33}(Al,Mg)_2(Si_4O_{10})(OH)_2{\cdot}nH_2O$ and was chosen for the ability for water to interact between octahedral and tetrahedral sheets causing weathering and release of cations into solution. It should also be noted that the crystal habit of montmorillonite is monoclinic prismatic as will be relevant during imaging of post treated bamboo. Citric acid was chosen as a weak organic acid and natural preservative to modify the pH of the resulting solutions.

3.0 Results

3.1 Raw Bamboo

It was critical to understand the properties of each bamboo starting material such that treatment results could be meaningful. Culm wall thickness, cross sectional area, density, moisture content, species, and age relationships are represented in Table II.

Table II - Bamboo starting materials and their meaningful properties relating to degradation and treatment

	Thickness (cm)	St. Dev.	Cross Sectional Area (cm^2)	St. Dev.	p (g/cm^3)	St. Dev.	Moisture Content (%)	St. Dev.
Henon (1 yr)	0.669	0.052	1.900	0.154	1.011	0.080	32.9%	5.5%
Henon (3 yrs)	0.498	0.065	1.285	0.151	1.055	0.234	21.3%	5.1%
Madake (1 yr)	0.854	0.123	4.799	0.773	0.954	0.202	34.7%	8.2%
Madake (3 yrs)	1.288	0.075	5.546	0.284	0.725	0.042	4.4%	3.4%

The two most critical properties are density, Figure 12, and moisture content, Figure 13.

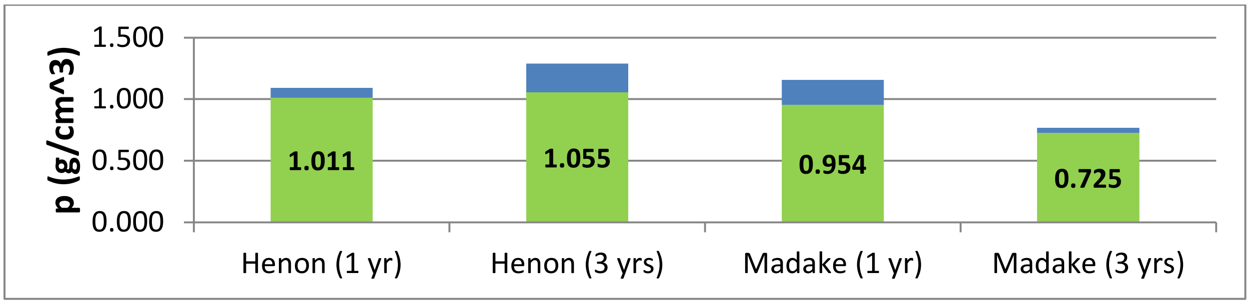

Figure 12. Density of different bamboo species at 1 year and 3 years old.

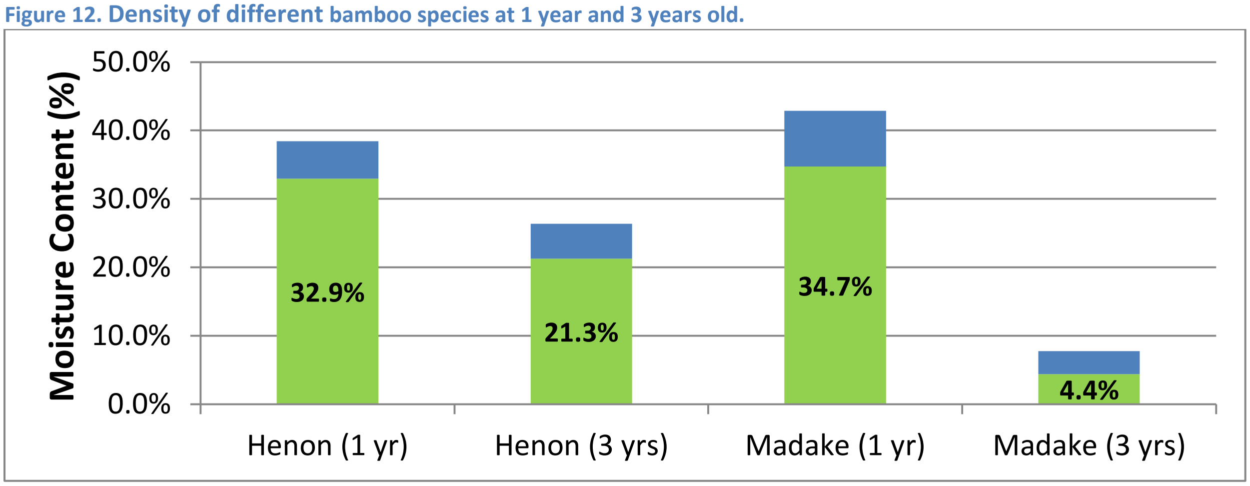

Figure 13. Moisture content of different bamboo species at 1 year and 3 years old.

In order to obtain absorption coefficients from the data collected it was necessary to linearize and normalize the graph shown in Figure 14.

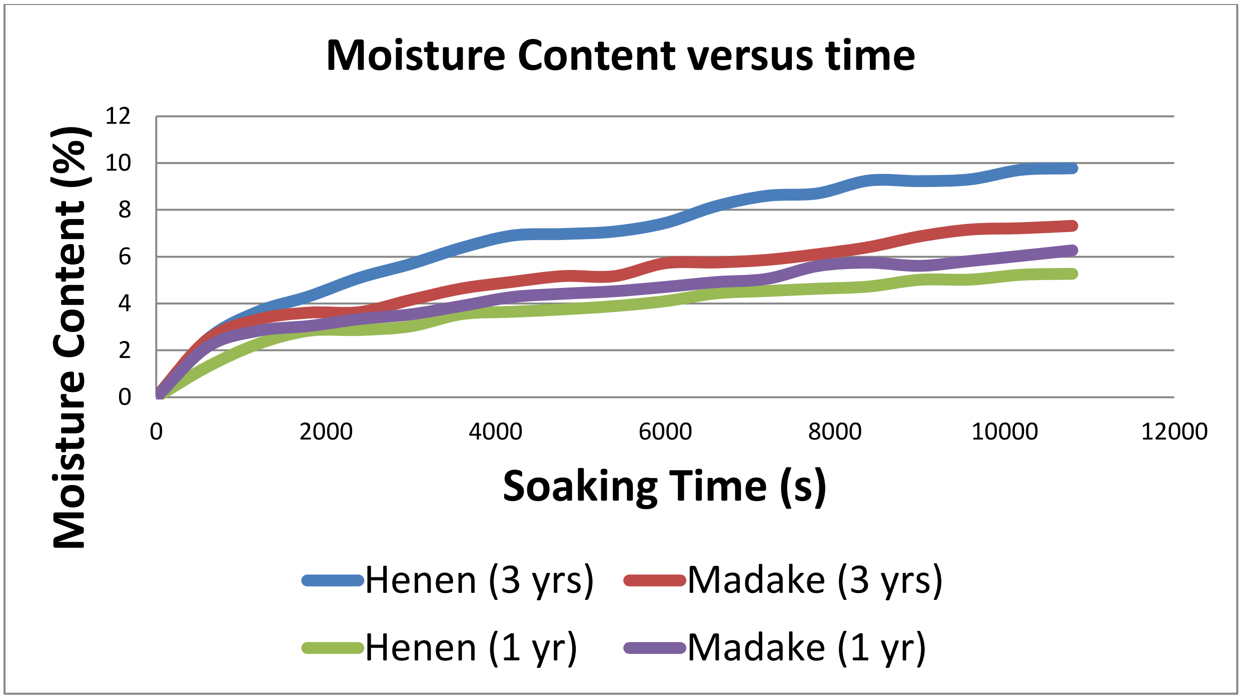

Figure 14. Moisture content (%) vs soaking time (s) for the raw bamboo samples

Figure 15 represents the graph normalized with respect to culm weight and the area of the culm exposed to the solution on the y axis with the square root of time on the x axis.

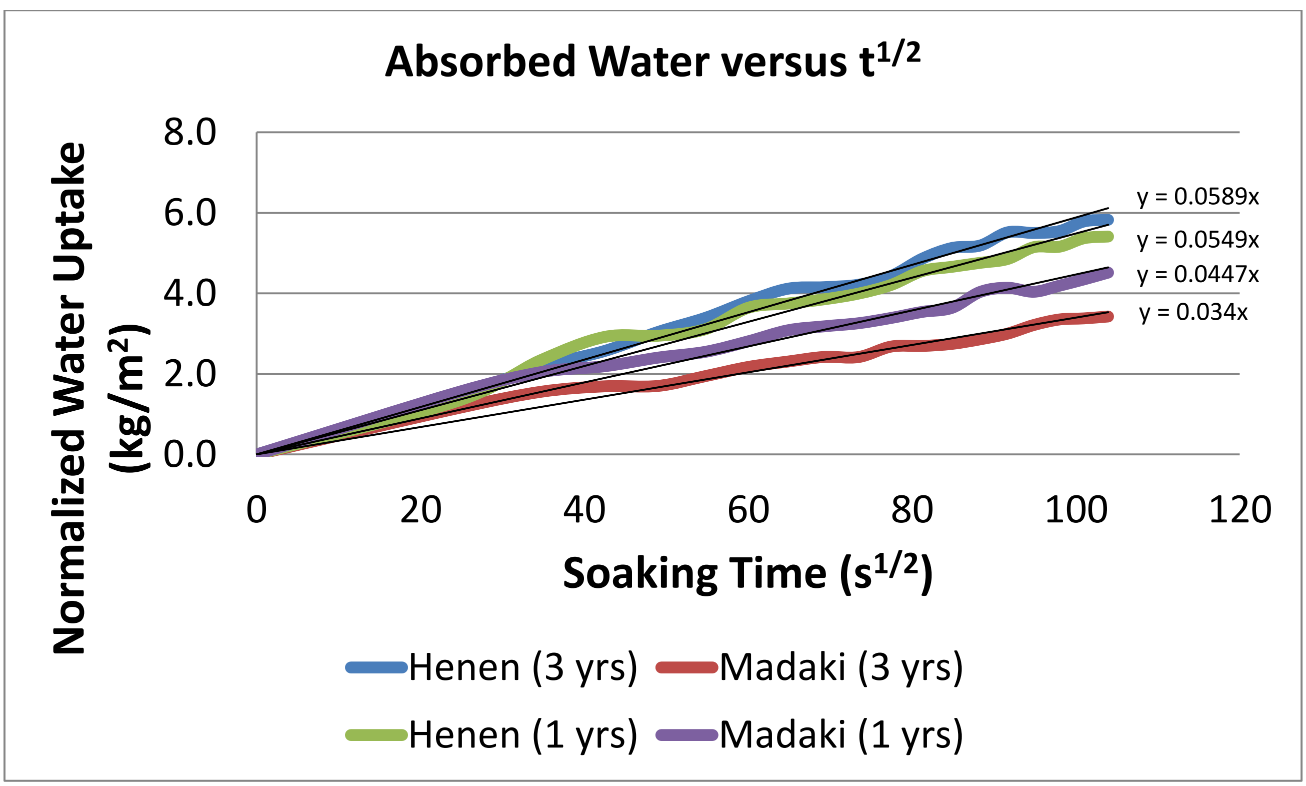

Figure 15. Normalized water uptake versus the square root of time in order to determine absorption coefficeints.

3.2 Treated Bamboo

The high variability and large sample quantity in thus study necessitated a reduction in variables and thus one solution was chosen for the rest of the study. Figure 16 shows images taken using ESEM of 3 year old Madake samples exposed to each solution for 2 hours.

Culms exposed to solutions of pH 6.5-7.3 showed limited mineralization; pH 8 evidenced mineral precipitations primarily within cell wells; and pH > 8.5 showed significant cellular decomposition and mineralization within cell pores. The solution of NaSi: M-H_2O: TCA in the ratio of 1.0: 120.0: 0.5 producing a pH 8 was chosen for the experiment for both treatment types based on results shown in Figure 17.

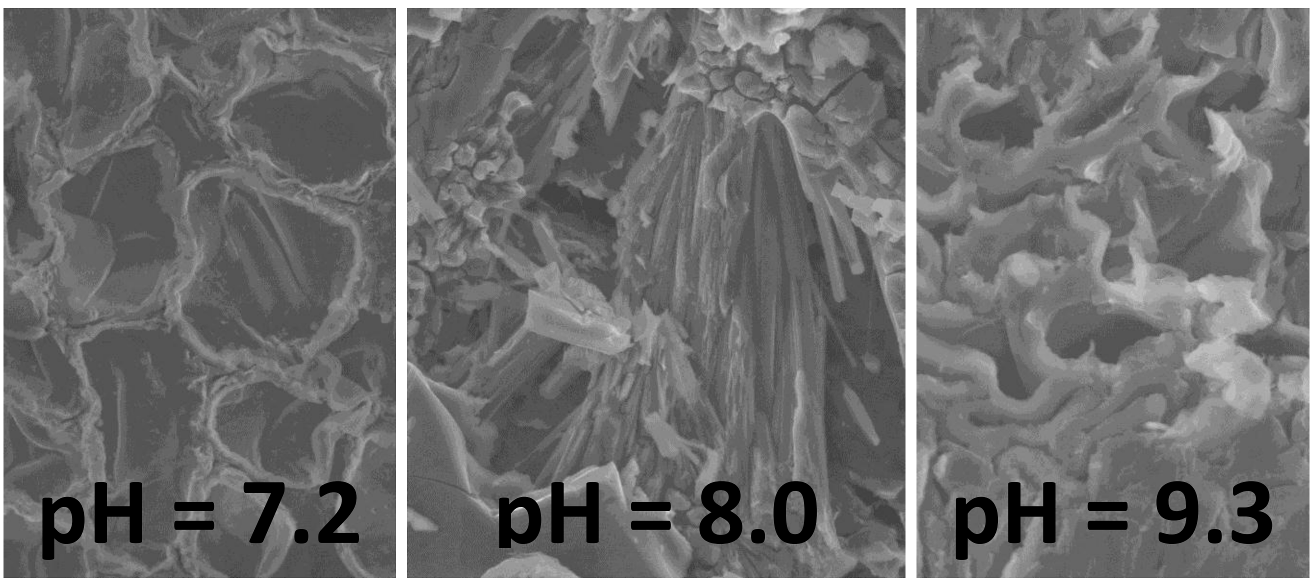

Figure 16. Environmental scanning electron microscopy of 3 samples taken at microscopy at 1000x, 10.8mm working distance, 20 kV showing the effect of pH on treatment success.

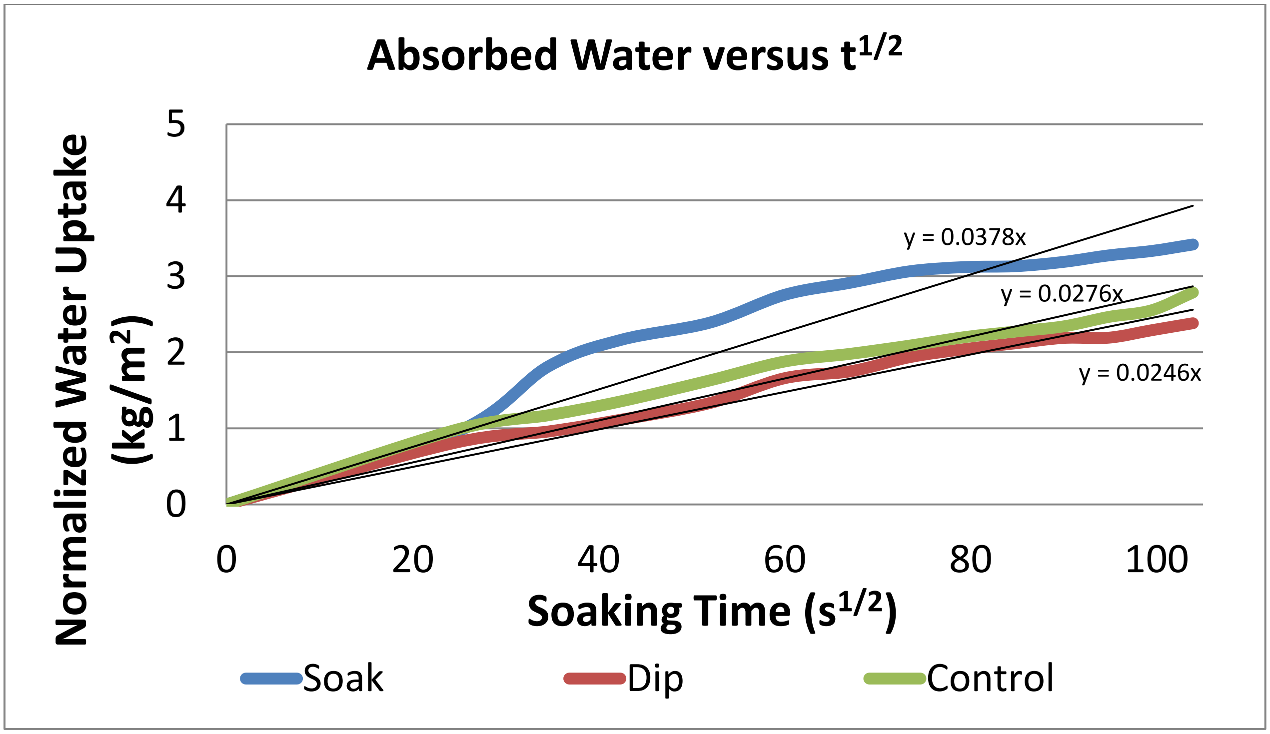

Figure 17. Normalized water uptake versus the square root of time in order to determine absorption coefficeints.

After this experiment additional dimensional measurements were taken and compared to their initial dimensions. The results can be seen in Figure 18.

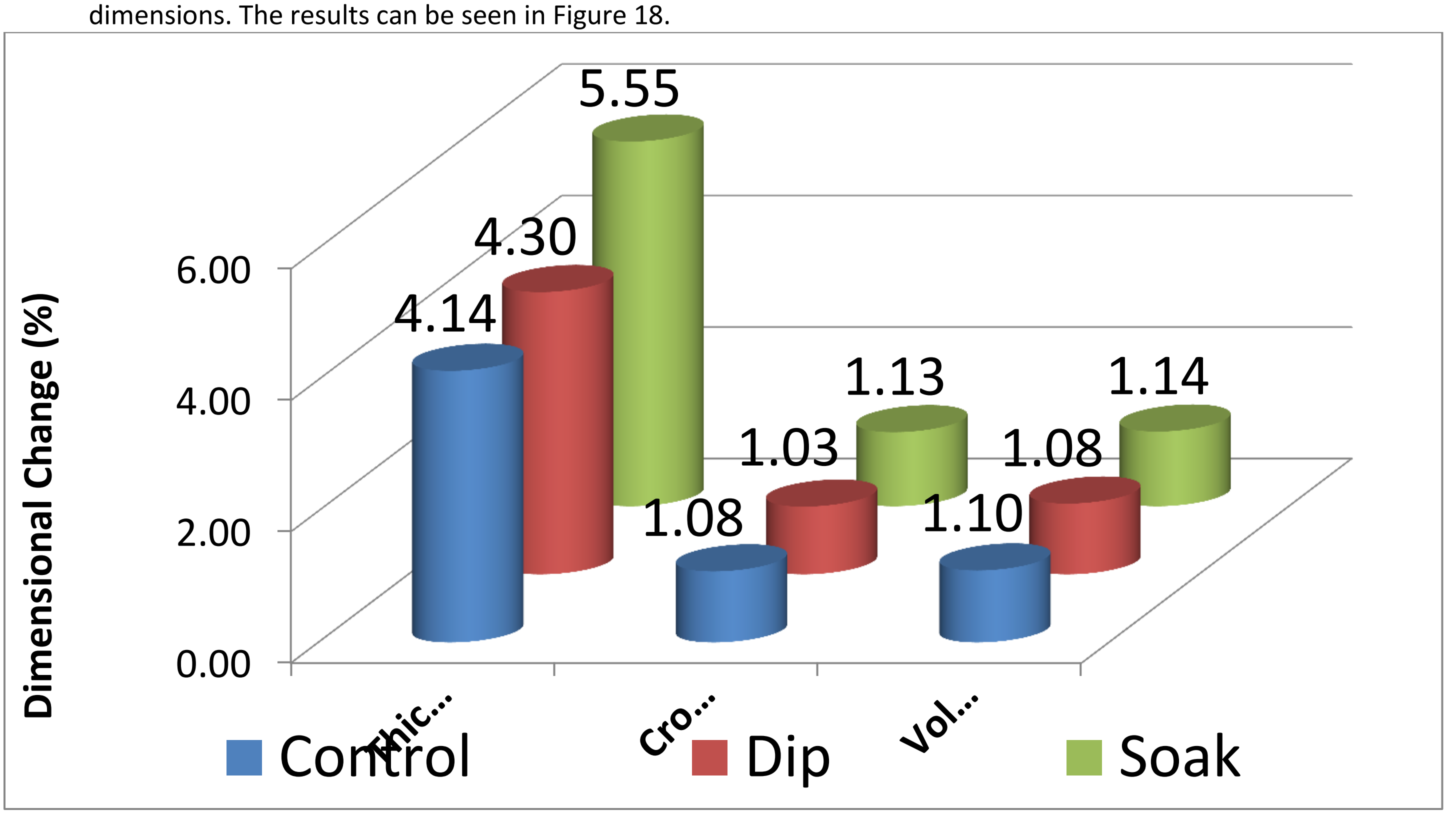

Figure 18. Percent Dimensional Change of thickness, cross-section, and volume for the control, dip, and soak treatments.

Lastly, XRD Analysis was used to identify the presence and chemical composition of precipitated minerals. The scans of the source materials as well as untreated and treated bamboo indicate the presence of albite, a sodium rich plagioclase feldspar, based on the peaks shown in Figure 19.

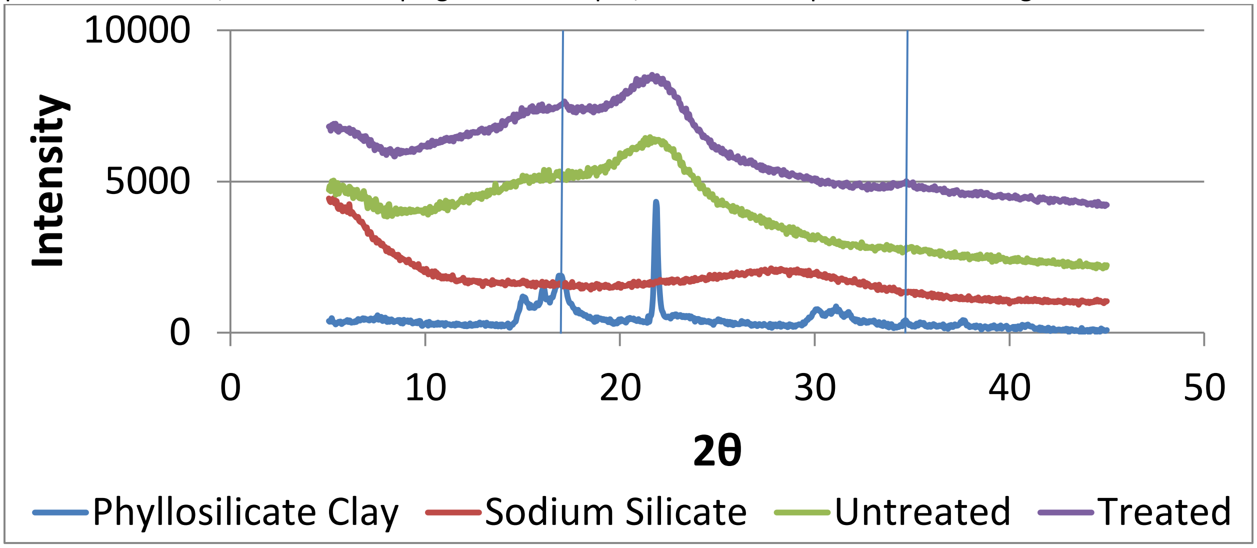

Figure 19. XRD analysis of treated/untreated bamboo and source minerals. The peak matchup corresponds to albite.

4.0 Discussion

4.1 Raw Bamboo

Literature references disagree on importance of using dry (aged) vs. wet (young) bamboo. Ghavami asserts that 'bamboo to be treated with a preservative necessitates the dry condition to facilitate penetration"[1]. Leise claims that the dry condition corresponds to a drying of cell sap that could possibly axially distort the structure as well as preventing penetration without preventing degradation [9]. In choosing the correct bamboo, Figure 15 illustrates that although age may play a factor in water absorbance, the result is different from species to species. For the Madake timber, older culms outperformed the young, while the opposite is true for Henen. The standardized relative moisture content test is based on mass gain and thus is inconsistent when measuring bamboos of different age and species. It is proposed that in addition to the leaching of lignin that occurring in all samples, hydrated cell sap diffuses out of young samples as a solution penetrates, thus negating some percentage of weight gain. It is clear that bamboo species type cannot be ignored due to an inherent difference in initial cell sap quantity.

If the absorbance numbers are instead graphed according to density, as in Figure 20, what is clearly visible is the influence of density in determining the rate at which bamboo absorbs water.

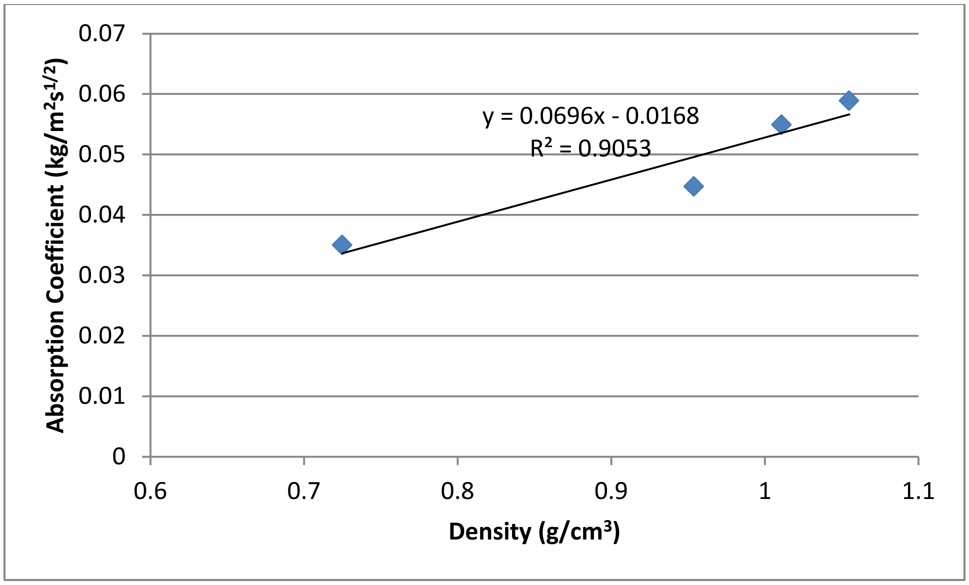

Figure 20. Absorption Coefficient vs. Density for bamboo types

This is likely due to the relationship between free and bound water in bamboo. Bound water is what binds between the cellulose and hemicellulose within cellulosic fibers and free water is the volume of water contained within the lignin based metaxylem vessels. Lower density bamboos have a lower density of reinforcing fibers and thus lower absorption coefficients. It is also important to note that the fibers are aligned axially and relate to the dimensional changes shown in Figure 18. These changes show axial dimension to be the least stable and thus most critical dimension to control. This knowledge could lead to an implementation strategy that inhibits axial swelling by fixing the bamboo ends against another structural member.

4.2 Treated Bamboo

The results are consistent with literature indicating that longer soaking times induce accelerated lignin leaching, but indicate that significant lignin leaching occurs within 24 hours. Soak diffusion treatments results are representative of significant lignin leaching during processing and correlate to a reduction in total bamboo hydrophobicity negating the effects of the mineralization treatment. Single dip treatments reduced the absorption coefficient of 3yr old Madake timber from 0.0276 to 0.0246. This is an 11% reduction in adsorption coefficient and it is hypothesized that successive dips will each have a reducing affect until a minimum absorption coefficient is reached.

XRD results indicate the retention of cellulose in dip treatments as well as the precipitation of albite minerals. Albite is a sodium rich feldspar with the formula, NaAlSi3O8. The precipitation of this mineral was not expected, but it makes sense considering an incredibly sodium rich source material such as

sodium silicate. It is suspected that there was a multitude of mineral precipitations that are masked by the broad cellulose peak. One method of extracting this data could be the removal of the cellulose matrix by acid and evaluation of the remnant minerals once dry. Another, more meaning, method would be utilization of micro-XRD to analyze the bamboo at a finer scale. This would provide the location of the each mineral phase which is expected to change based on nucleation location. This would also allow for a better understanding of the latent silica content within the bamboo, especially in the silica rich cortex.

5.0 Conclusion

1. Selection of bamboo species plays a significant role in the determination of water absorption and age relationships may be different from species to species.
2. Less dense bamboo has a lower absorption coefficient due to a difference in bound vs. free water.
3. Soaked bamboo experiences significant lignin leaching exposing more hydrophilic cellulose and increasing the absorption coefficient.
4. Dipping treatments utilize evaporative precipitation and have shown a 11% reduction in absorption coefficient of bamboo.

6.0 Recommendations

- In order to determine the maximum effectiveness of treatment, the time dependence of lignin leaching in cellulose materials should be further studied.
- Successive dip treatments should be investigated to determine a maximum reduction in absorption coefficient.
- Mineralized bamboo species should be tested within a concrete system to determine the effectiveness compared to untreated bamboo as well as non-fixing, non-toxic treatments and fixing, toxic treatments.
- Micro-XRD should be used to identify the exact location of amorphous cellulose, crystalline cellulose, and mineral precipitations.

7.0 References

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