



Quantifying the viscoelastic properties of switchgrass using micro/nano-engineering tools : preliminary results

Acknowledgment

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Objective

- Study the effect of genetic modification and enzymes on the mechanical properties of the switchgrass

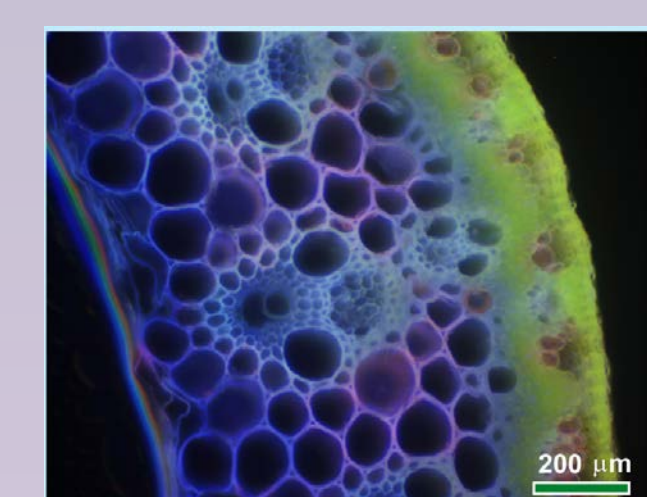
- Distinguish between switchgrasses with different amount of cellulose to lignin ratio. using indentation techniques.
- Determination of material properties of switchgrass using indentation techniques

Introduction

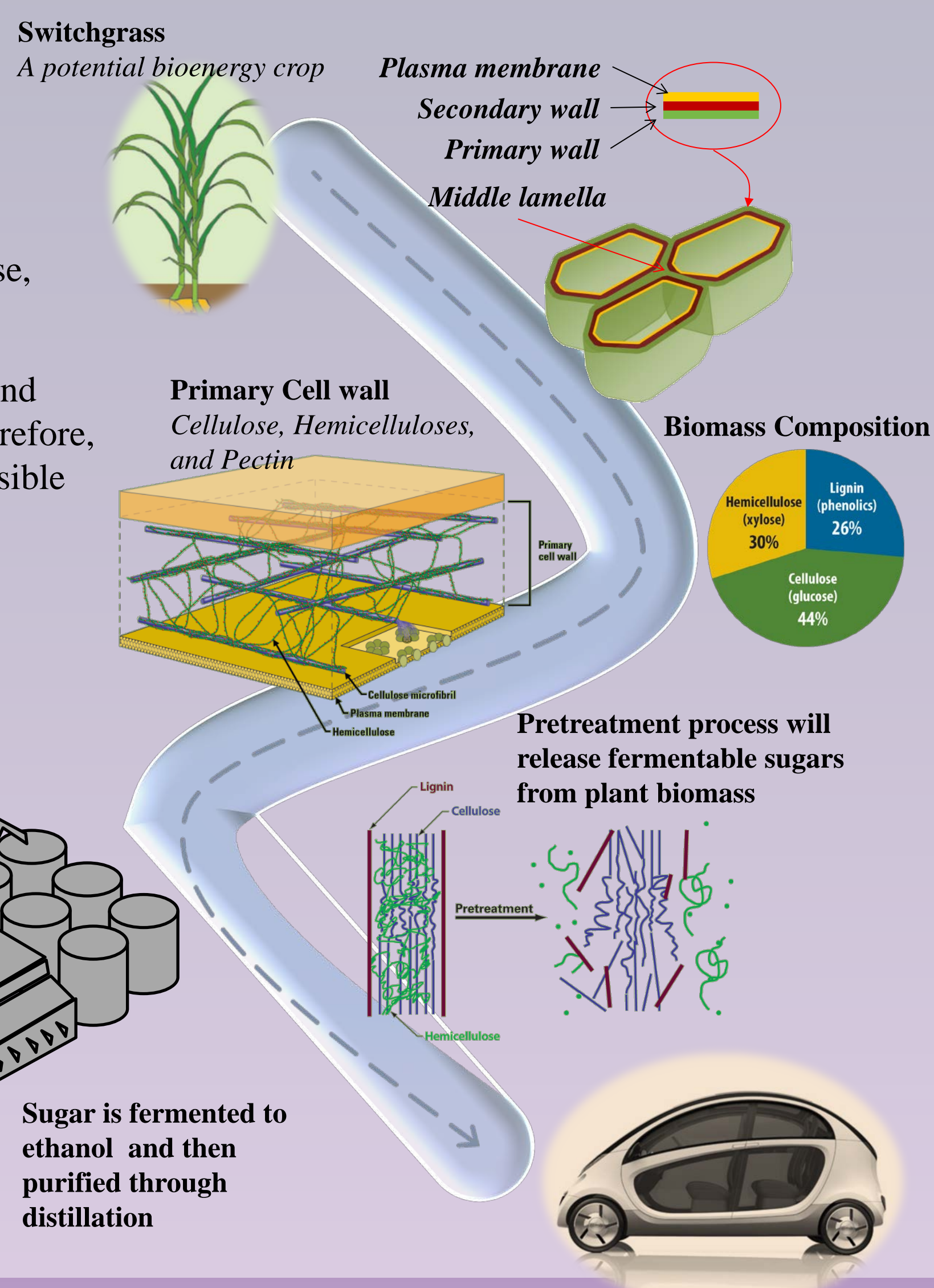
Biotechnology for biofuel

Biotechnology offers the possibility of increasing ethanol producing using cellulose, and other polysaccharides.

Biofuel is mostly produce from cellulose and hemicelluloses component of the wall. Therefore, it is essential to make cellulose more accessible to enzymatic breakdown (hydrolysis) and solubilizing hemicellulose sugars.



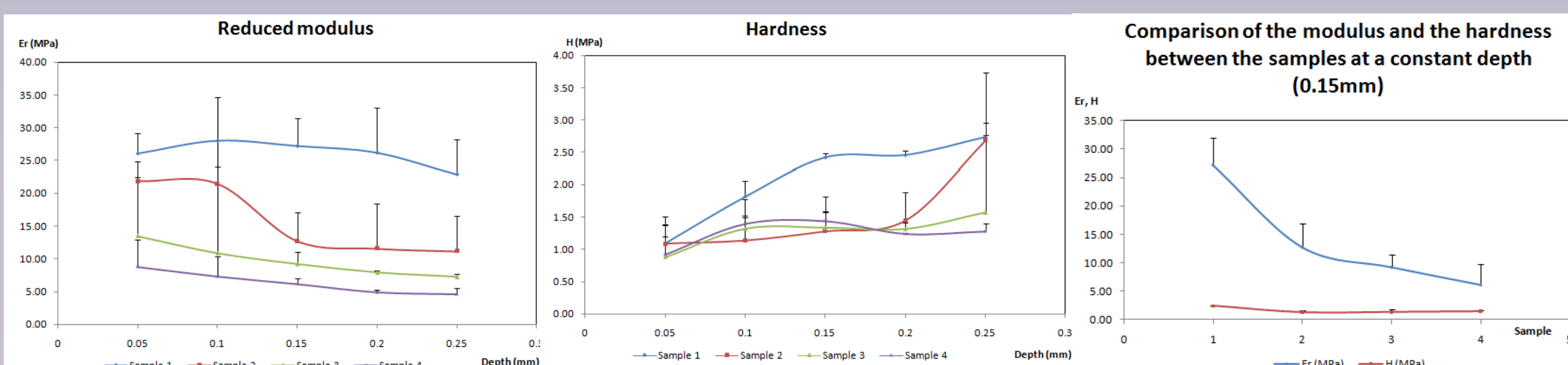
Deeper blue areas toward the stem's center show more highly lignified xylem tissue used for water and nutrient transport. Therefore, there should be a difference in mechanical properties along the radius.



Results and discussion

Microscale test results

The elastic modulus is obtained from **micro-indentation** at different depth and at specific location on the stem.



By testing different parts of the stem, one can see that the modulus is dependent to the location of the indentation. The modulus of the samples 1, 3 and 4 are quite constant with the depth whereas the modulus of the sample 2 decreases after a 0.15mm-depth.

The hardness can be also calculated from the same test. In this case, the sample also has the most significant behavior. The value is increasing, especially after 0.2mm. This increase can be due to the change of behavior between the cellulose (exterior layer) and the lignin.

Depending on the location on the stem, the modulus is changing whereas the hardness is constant. The decrease of the modulus can be due to a loss of lignin on the highest part of the stem.

From these preliminary results, stiffness and hardness decrease with height on the plant corresponding to our expectations : more lignin near the bottom and more cellulose near to the top of the plant.

Nano scale test results

The relaxation modulus curve versus time was obtained from load versus time curve. Then relaxation modulus versus time was calculated. Samples did not show any significant differences in their viscoelastic behavior, therefore, a different approach should be suggested for testing the samples (such as testing the cross section of the samples) in nanometer scale.

Experiment

Quasi Static indentations

The experiments were performed on five sections of switchgrass stem in quasi static mode. The test can be performed:

- Over a constant load and in an accurate point
- Displacement or load control indentation

From the force displacement curves reduced modulus and hardness of the material can be obtained using the following equations:

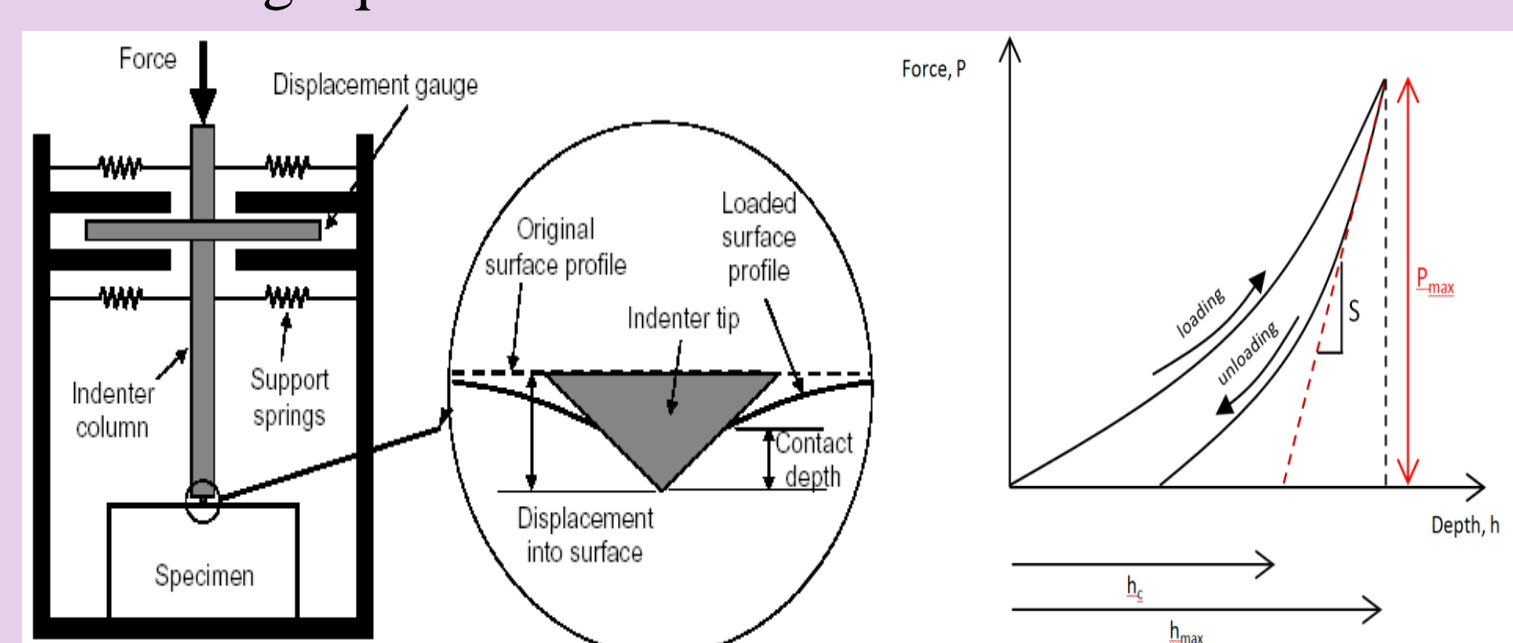


Diagram of indentation system and an indentation curve h_c : final depth h_{max} : depth corresponding to the maximum load

$$E_r = \frac{S \sqrt{\pi}}{2 \sqrt{A}}$$

S: stiffness, slope of the unloading curve
A: area, obtained by using the function area and the value of h_c

$$H = \frac{P_{max}}{A}$$

P_{max} : maximum load
A: area, obtained by using the function area and the value of h_c

Micro-indentation tests

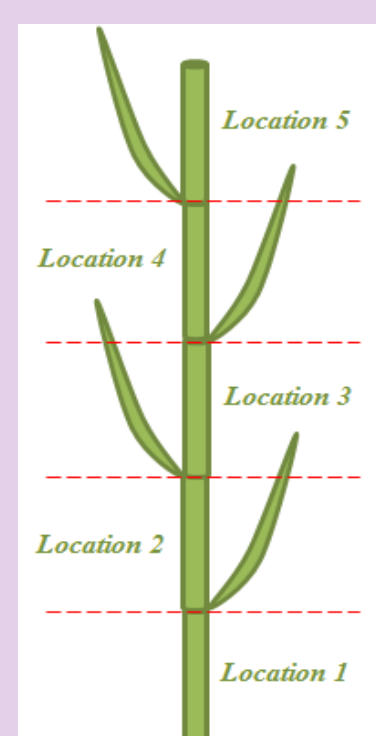
Micro indentations were performed using a Bose Electroforce 3200[®] in quasi static mode.



Bose Electroforce

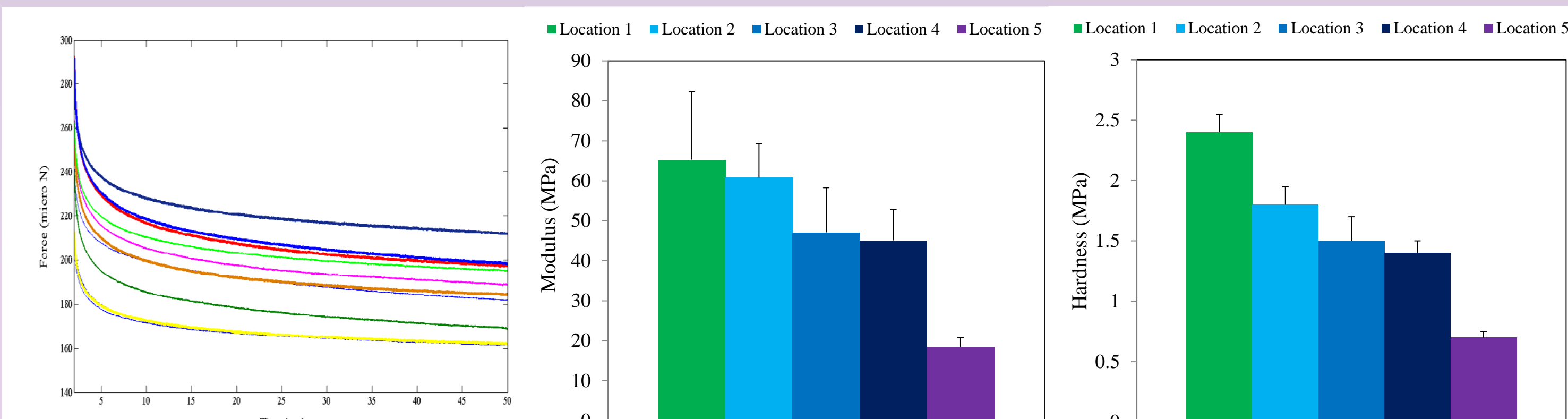
According to our hypothesis the lignin content in the sample should have a direct effect on the mechanical properties. Therefore, the indentations were performed at different depth as well as different locations along the stem of the switchgrass to study the mechanical properties.

Five locations were selected for measurements, with Location five closer to the top.



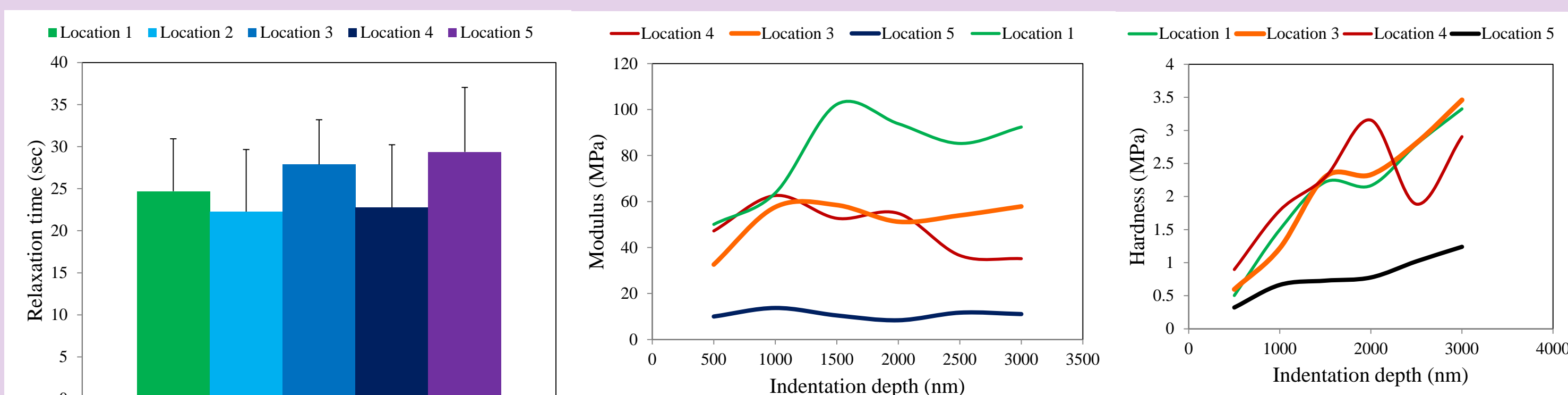
Nano indentation tests

Nano indentation tests were performed using a Hysitron Bio-Ubi[®]. A standard conical tip with a radius of 3µm was used as the indenter. Relaxation tests (at 1000 nm) were performed to obtain the viscoelastic properties and from load unload experiments (at 1000 nm) the modulus and hardness of the samples were quantified.



Relaxation test results on Location 3 performed using nano-indentation technique. The reproducibility of the results is reasonable.

Reduced modulus and hardness results shows a decrease in value along the stem of the switchgrass, showing less stiffness and hardness towards the top of the stem, indicating less lignin and more cellulose content.



The results obtained from relaxation tests were almost the same for all the locations.

Indentation at different depth were performed, up to 3µm depth and modulus and hardness values were calculated.

Conclusion and future work

- Micro indentation seems to be a good method to compare the mechanical properties in different parts of a switchgrass plant. the modulus and hardness values will be related to the lignin content in each sample.
- Nano indentation results follow the same trend and show higher mechanical properties closer to the base of the plant, however, as mentioned earlier up to a certain depth, around 200µm, cellulose is the dominant component and after that lignin is the main component.

References

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- Chapple C, Ladisch M, Meilan R, Loosening lignin's grip on biofuel production. Nature Biotechnology Vol.25, No 7 (2007)
- Kristensen JB, Thygesen LG, Felby C, Jorgensen H, Elder T. Cell-wall structural changes in wheat straw preheated for bioethanol production. Biotechnology for biofuels 2008, 1:5.