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Nanoindentation characterization of microwave-pyrolysis biochar

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Nanoindentation Characterization of Microwave-Pyrolysis Biochar

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Biochar II: Production, Characterization and Applications

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Research Motivation & Goals

Strong potential for biochar in biocomposites manufacturing

- Can aid in compressive and flexural strength
- Can increase flammability resistance

- Need a better understanding of biochar mechanical properties

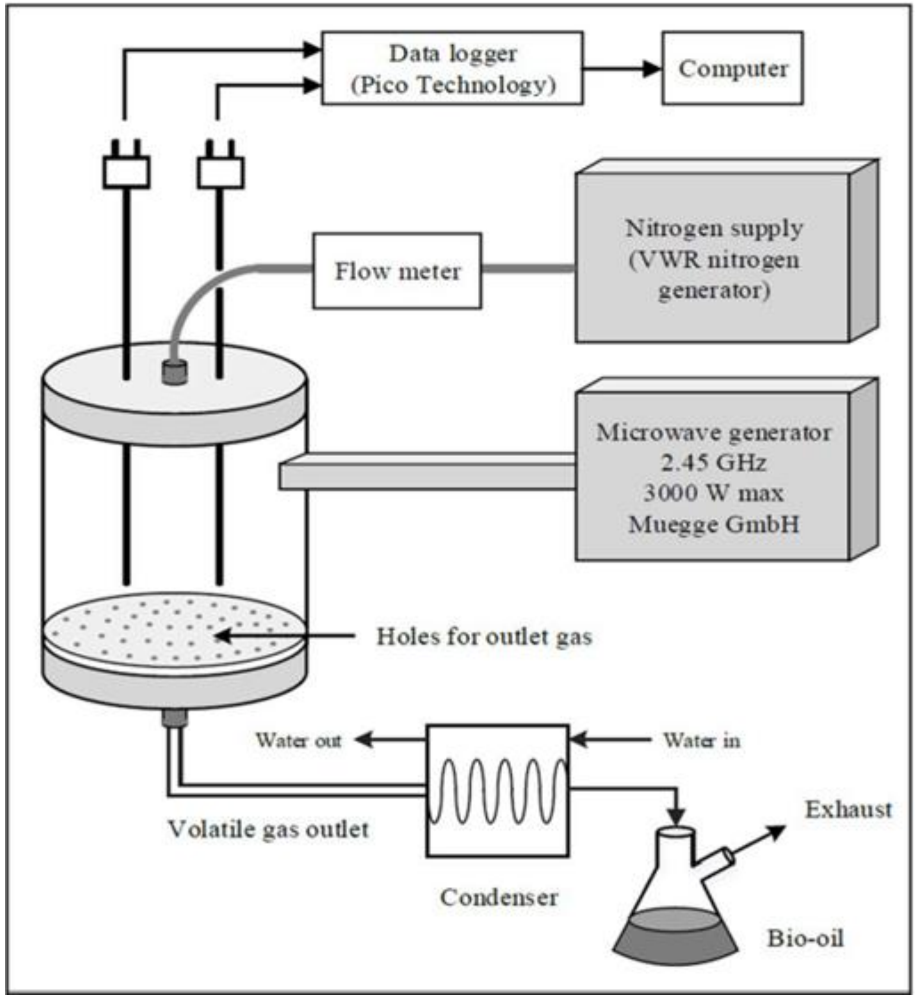
- Goals
  - Produce biochar via microwave pyrolysis
  - Characterize biochar through common techniques and nanoindentation
  - Evaluate the Young’s modulus and hardness of char

Research Methodology – Feedstocks

- Softwood Chips
  - Mixture of spruce/fir
  - Non-uniform sizes
  - Moisture content ~12%
- Hemp
  - Dried Bale
  - Hand shredded, Non-uniform sizes
  - Moisture content ~13%
- Both feedstocks were locally sourced

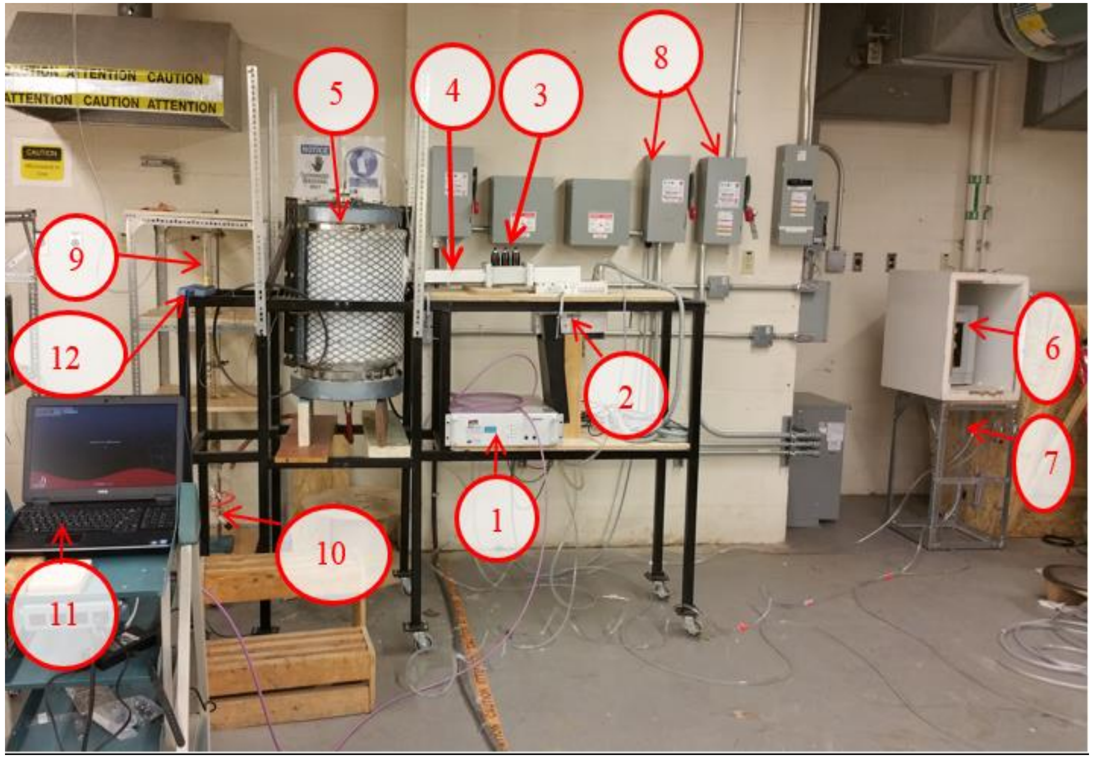
Research Methodology – Microwave Pyrolysis Reactor

- Experimental parameters
  - 1 kg, feedstock
  - 100 grams, carbon microwave absorber
  - 1 hr run time
  - 2700-Watt microwave power level



Scaled-up microwave reactor, UNB

- 3000 Watt max
- Large insulated single batch reactor
- Real time temperature data
- Volatiles exit through condenser
- Max. 5 kg sample



MW power supply (1), Magnetron head (2), Sub-tuner (3), Waveguide (4), SS 309 reactor (5), N_2 gas generator (6), Flow meter (7), Main power switch (8), Condenser (9), Bio-oil collector (10), Computer and data logging system (11), Temperature data acquisition system (12)

Salema, A.A., and **Afzal, M.T*.,** and Bennamoun, L. 2017. Pyrolysis of corn stalk biomass briquettes in a scaled-up microwave technology. ***Bioresource Technology,*** 233:353-362.

Research Methodology – Biochar Characterization

Biochar Characterization

1) Ultimate and Proximate analysis
2) Physiosorption analysis
3) SEM imaging
4) Nanoindentation

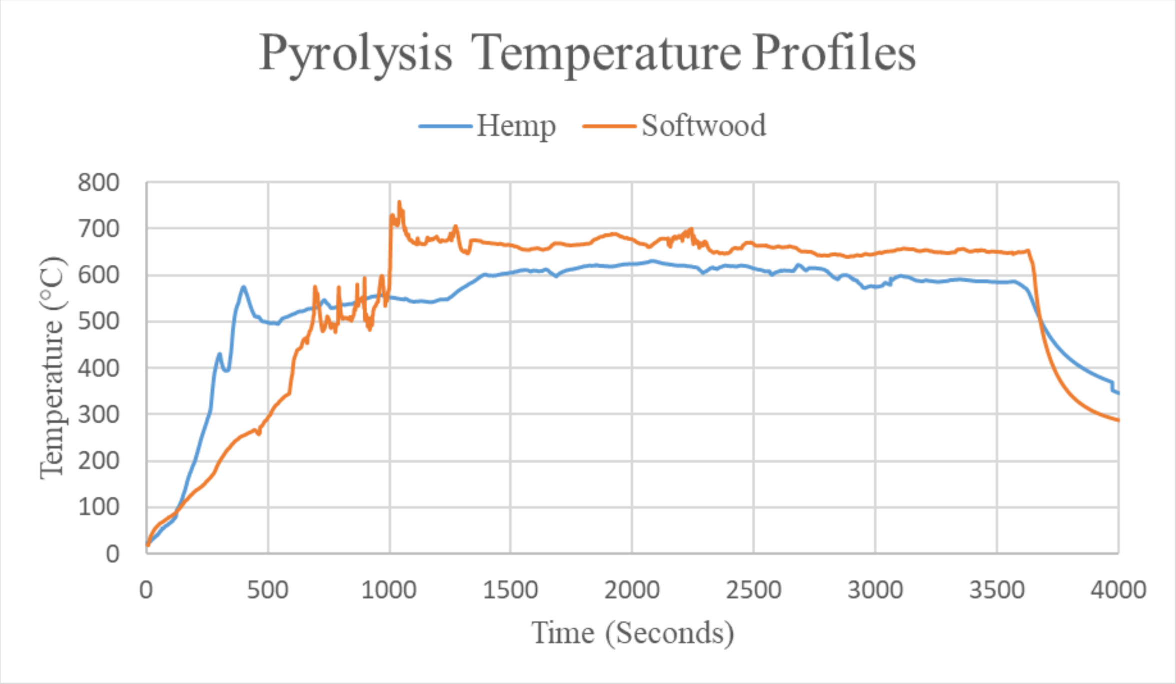
Research Methodology – Biochar Characterization

Nanoindentation

1) Cold-mounting samples
2) Polishing with decreasing grit sizes (300-1200 microns)
3) Testing with iMicro Nanoindentor (10 indentations per sample)

nanomechanicsinc.com

Results – Microwave Pyrolysis Temperature



Feedstock	Residence Temperature (°C)	Heating Rate (°C/min)
Softwood	659.8 ± 59.9	38.7 ± 13.3
Hemp	604.2 ± 3.3	49.2 ± 10.8

Results – Biochar

<u>Softwood</u>

<u>Hemp</u>

Results – Biochar Proximate/Ultimate

	Proximate Analysis				Ultimate Analysis			
Specimen	M.C. %	V.M. %	Ash %	F.C. %	Carbon %	Hydrogen %	Nitrogen %	Oxygen %
Raw Hemp	10	76	2	22	44.43	6.16	0.18	49.23
Raw Softwood	10	78	1	21	45.71	5.89	0.00	48.40
Hemp Char	3.0	26.8	1.0	72.2	78.5	3.3	0.6	17.6
Softwood Char	4.0	26.0	2.1	71.9	77.5	3.6	0.1	18.8

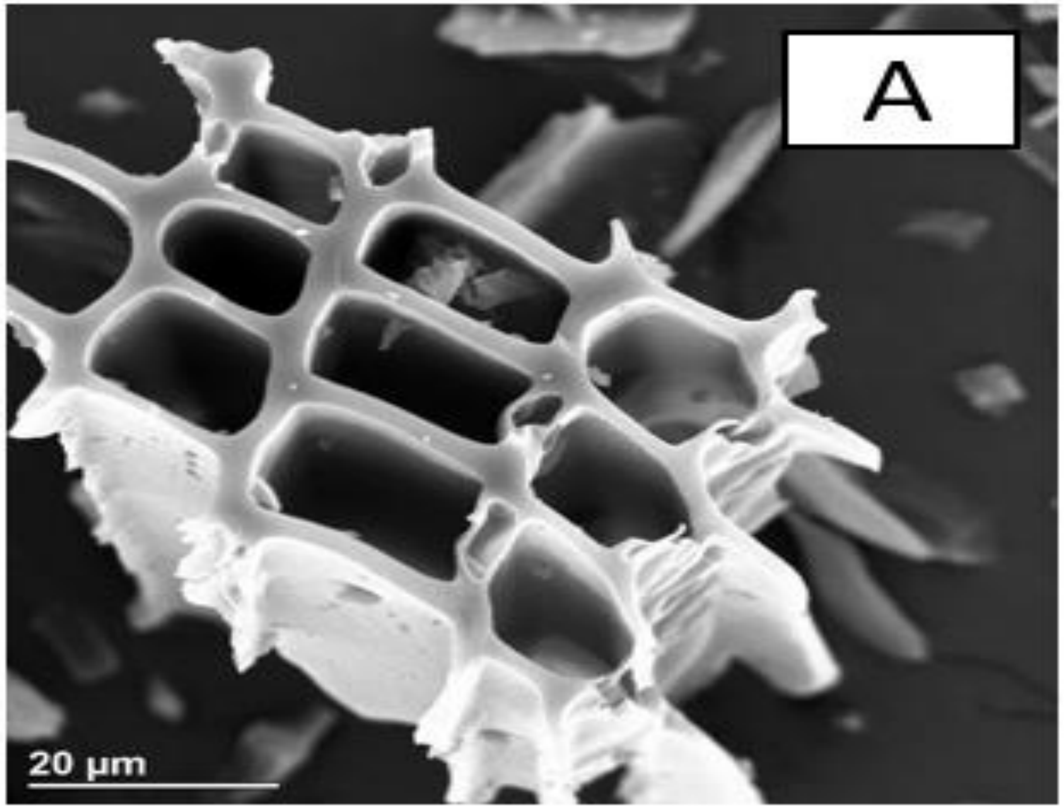
- Large increase in Fixed Carbon % and Carbon elemental % after pyrolysis due to release of volatiles
- Hemp biochar has larger carbon weight percentages than softwood (more full release of volatiles)

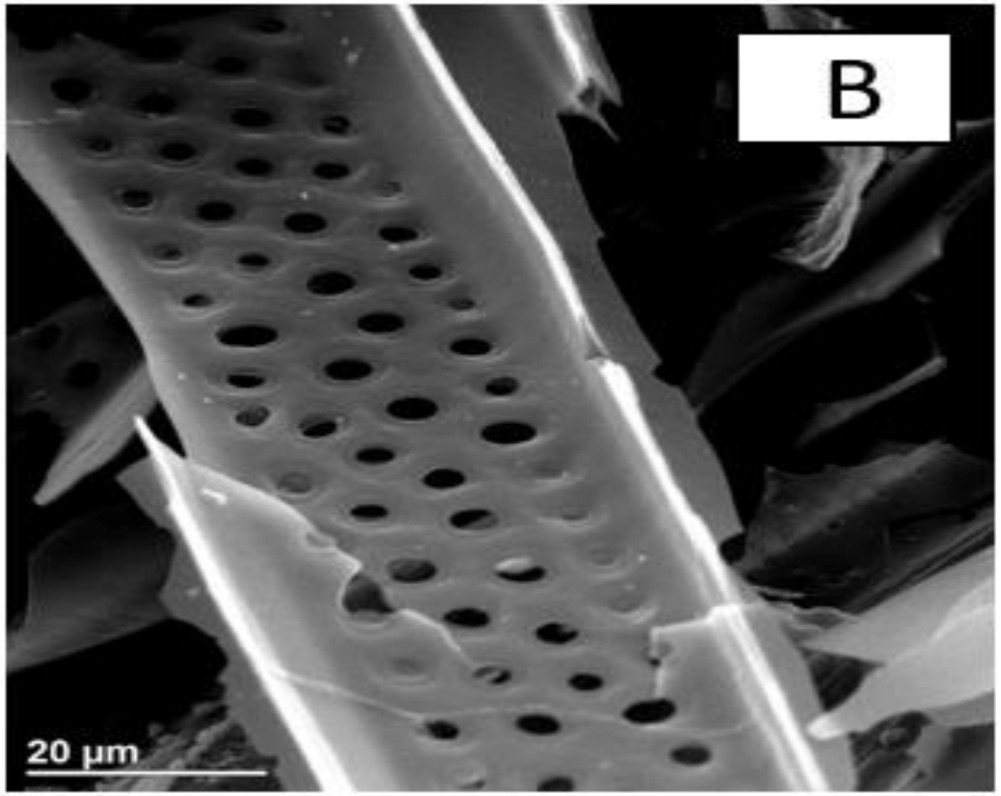
Results – Biochar Porosity

Sample	BET Surface Area (m^2/g)	Average Pore Diameter (Å)	Micropore Area (m^2/g)	Average Pore Volume (cm^3/g)
Raw Hemp	2.97	78.06	N/A	0.0058
Raw Softwood	0.76	134.86	N/A	0.0026
Hemp Char	12.18	52.75	2.58	0.0161
Softwood Char	9.96	46.44	1.63	0.0116

- Increase in BET Surface Area and Micropore Area due to development of new micropores during pyrolysis
- Hemp Biochar has better porosity properties due to faster heating rate allowing a faster release of volatiles

Results – Biochar SEM softwood (A) and hemp (B)

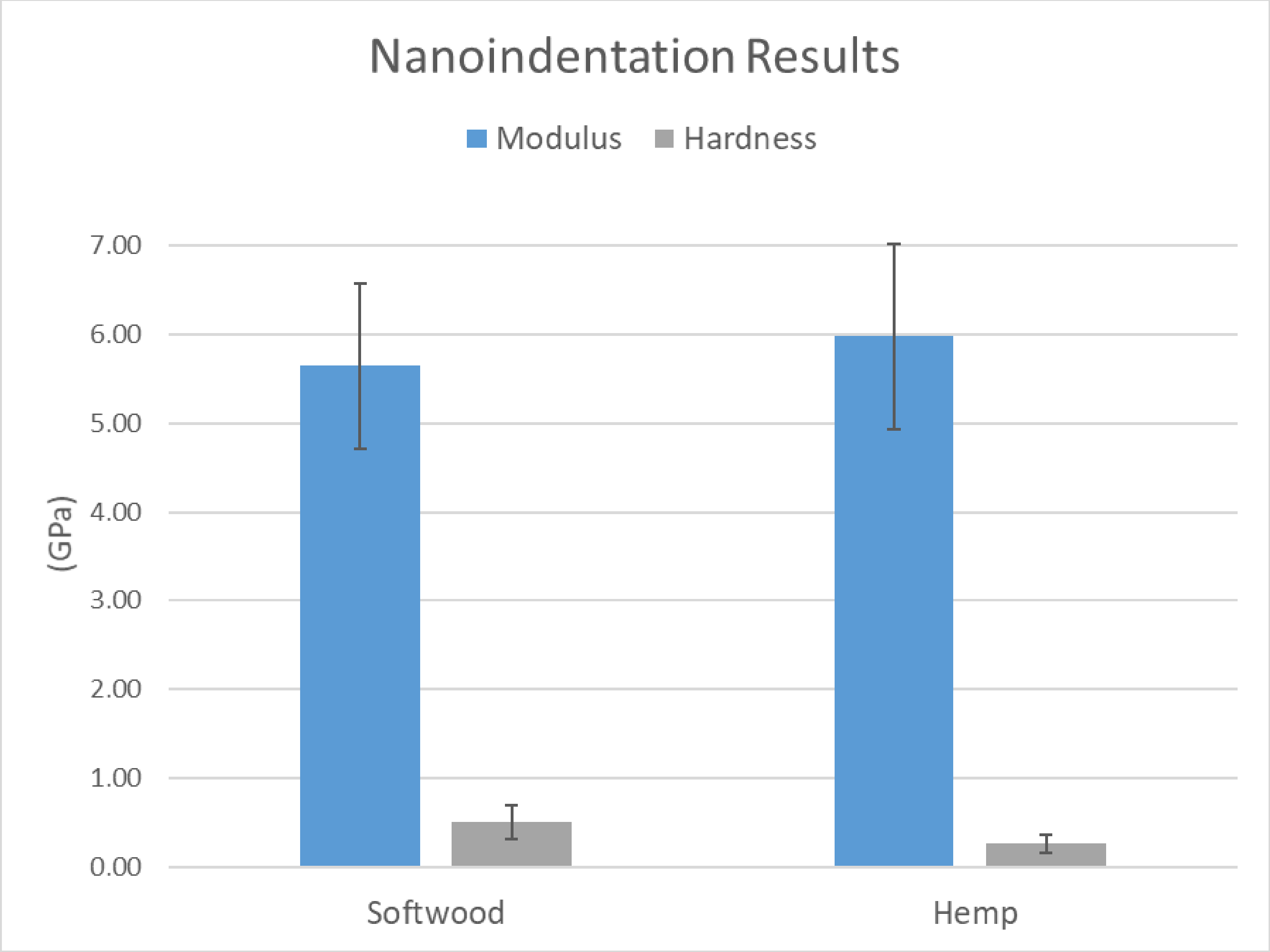




Images taken at 1000x magnification. Power level 27kW

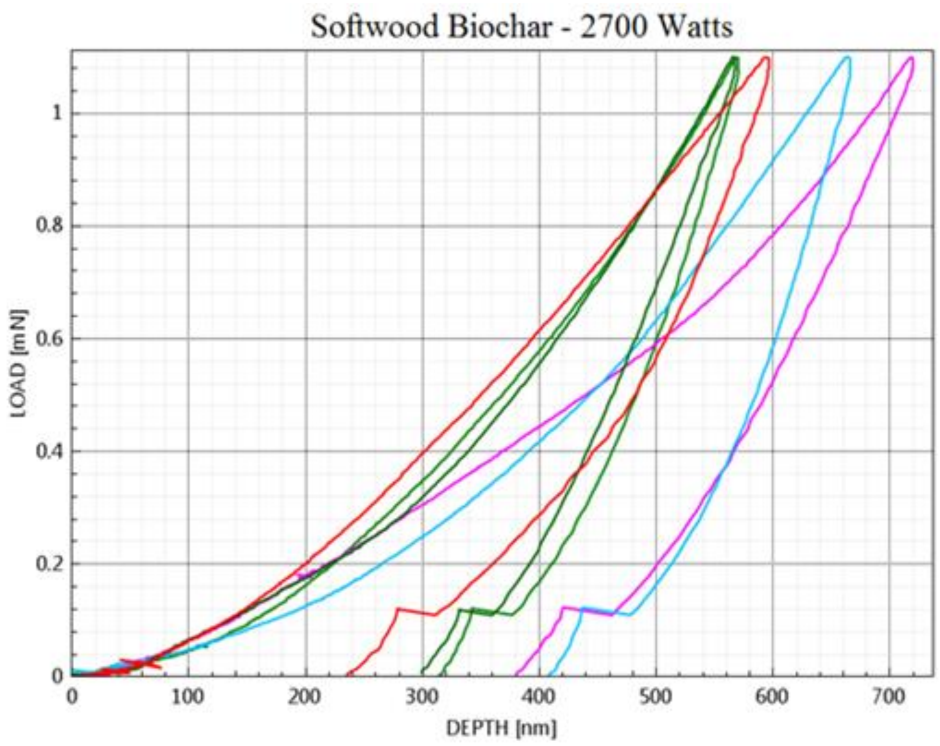
Wallace, C.A., **Afzal, M.T*.** & Saha, G.C. Bioresour. Bioprocess. (2019) 6: 33.

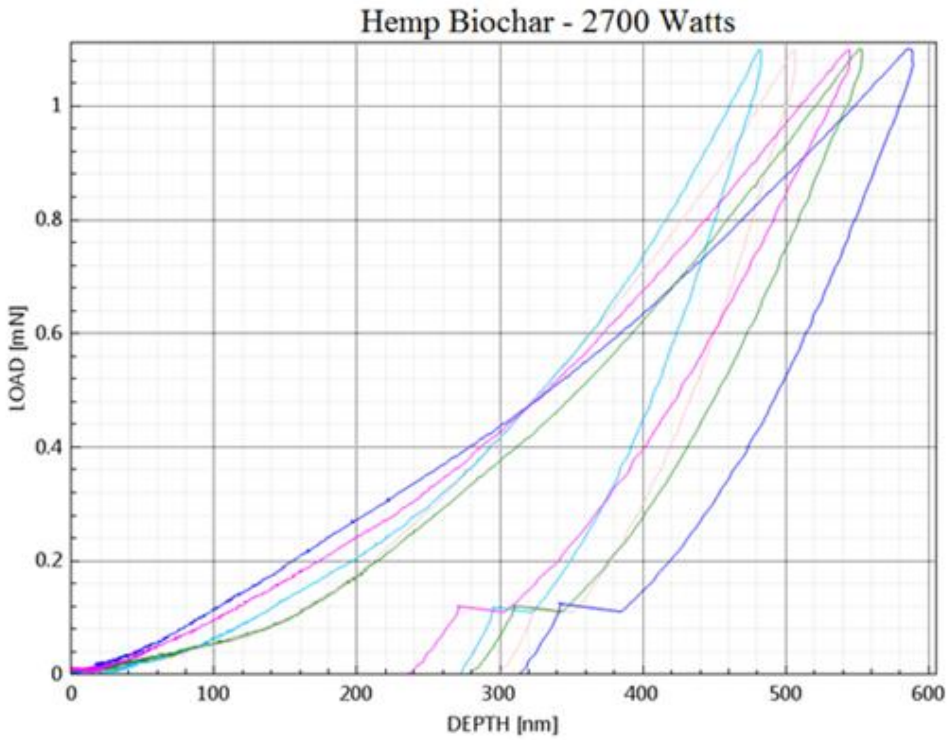
Results – Biochar Nanoindentation



Hemp biochar has a higher Young's modulus (resistance to deformation)

Results – Biochar Nanoindentation





Both biochar show partially elastic load-depth curves

Wallace, C.A., **Afzal, M.T*.** & Saha, G.C. Bioresour. Bioprocess. (2019) 6: 33.

Conclusion

- Hemp biochar showed a higher average Young’s Modulus than the softwood char
- Young’s modulus and hardness of biochar are dependent on the amount of carbon percentage and development of healthy micropore lattice
- Both samples would be suitable for biocomposite manufacturing due to their high Young’s Modulus values

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Acknowledgements

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THANK YOU!
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