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Biochar: From ligno-cellulosic materials to engineered products for environmental services

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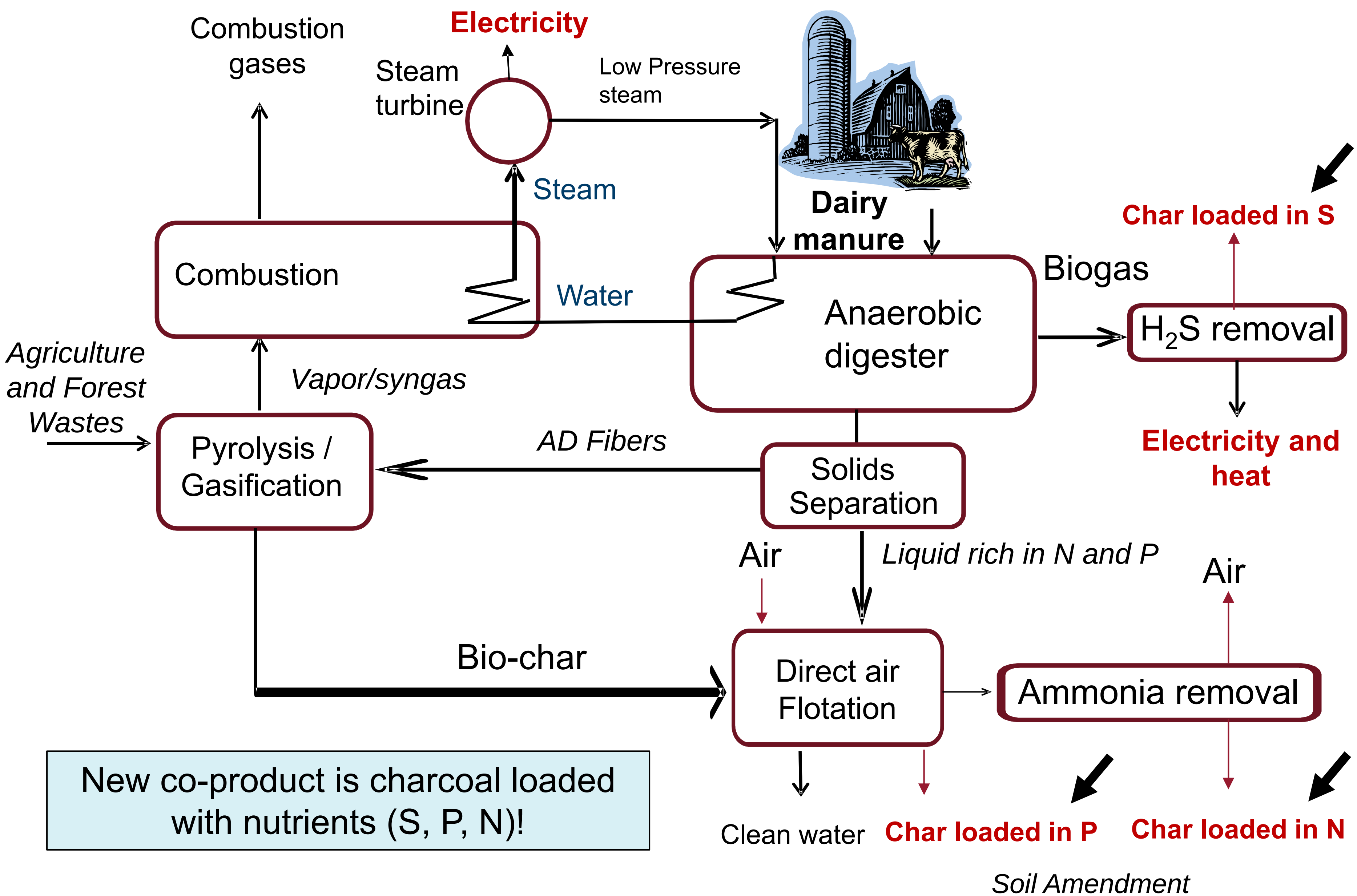
From Lignocellulosic Materials to Engineered Products for Environmental Services

Manuel Garcia-Perez, Matt Smith, Waled Suliman

Conference: Bio-char: Production, Characterization and Applications

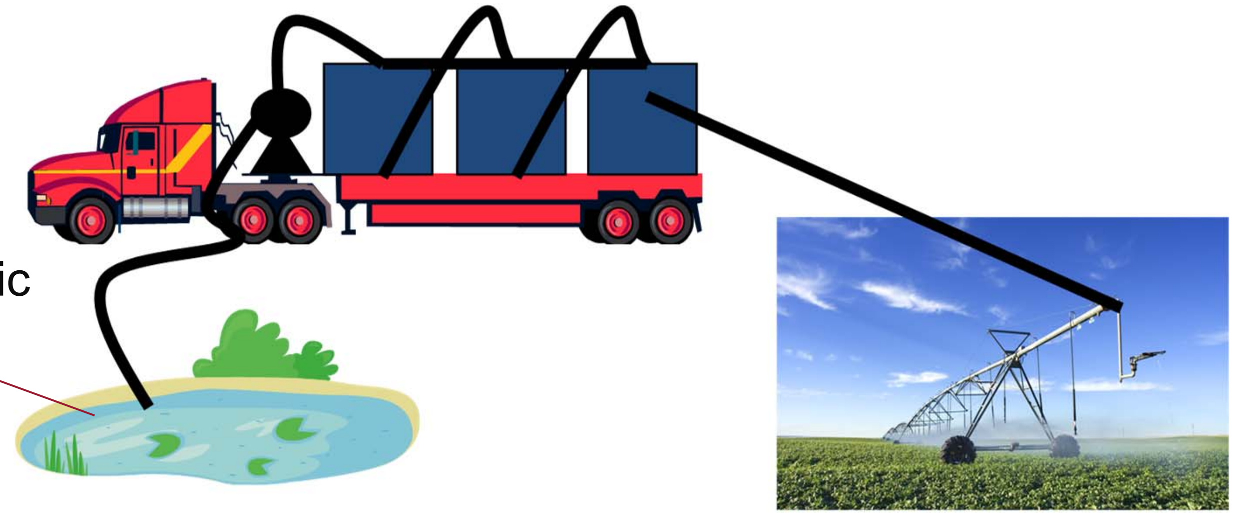
Engineering Conference International
August 20-25
Hotel Calissano, Alba, Italy

Introduction (WSU AD bio-refinery concept)



Introduction

N, P, E-coli, Organic Pollutants



Development of Cheap Engineered Bio-chars for Nutrient Removal

Ammonium Removal: Air stripping and ammonia collection in gas phase

Phosphate Removal: Colloidal Phosphorous Filtration and Phosphate ions precipitation on Ca and Fe

E-coli Retention: Adsorption on Positively Charged Bio-chars

Removal of Organic Pollutants: Adsorption on high surface area materials (Physical Adsorption)

Introduction

Strategy to Develop Engineered Bio-chars for Anerobic Digestion Biorefineries

Understanding the relationship between biomass composition and processing conditions on the formation of carbonaceous materials with functionalities (surface area, cation exchange capacity, surface charge, presence of Ca, and Fe on the surface) relevant to the removal of nutrients on AD effluents

↓

Development and Evaluation of Engineered Bio-chars at Laboratory Scale

↓

Production of Larger quantities of Bio-chars for Field Testing

↓

Field Testing

↓

Commercialization

Social impact increases, Research and Development Costs augment dramatically, Capacity to train student and Publish in Peer Reviewed Journals decreases

Introduction

Environmental Pollutants

Organic Molecules: Catechol, Phenol, Sulfamethazine

Heavy Metals: Pb (II), Cu (II), Cr (VI), Zn (II)

Inorganics: Phosphate, nitrates

Gases: CO, CO_2, NO, CH_4

Mechanisms for the Removal of Environmental Pollutants

Pore Filling

Diffusion and partitioning

Formation of surface complexes

Aromatic π-π interactions

Hydrogen bonding

Electrostatic interactions

Interaction with amine groups

Precipitation

Hydrophobic interactions

Cation Exchange

Simultaneous adsorption/catalytic degradation

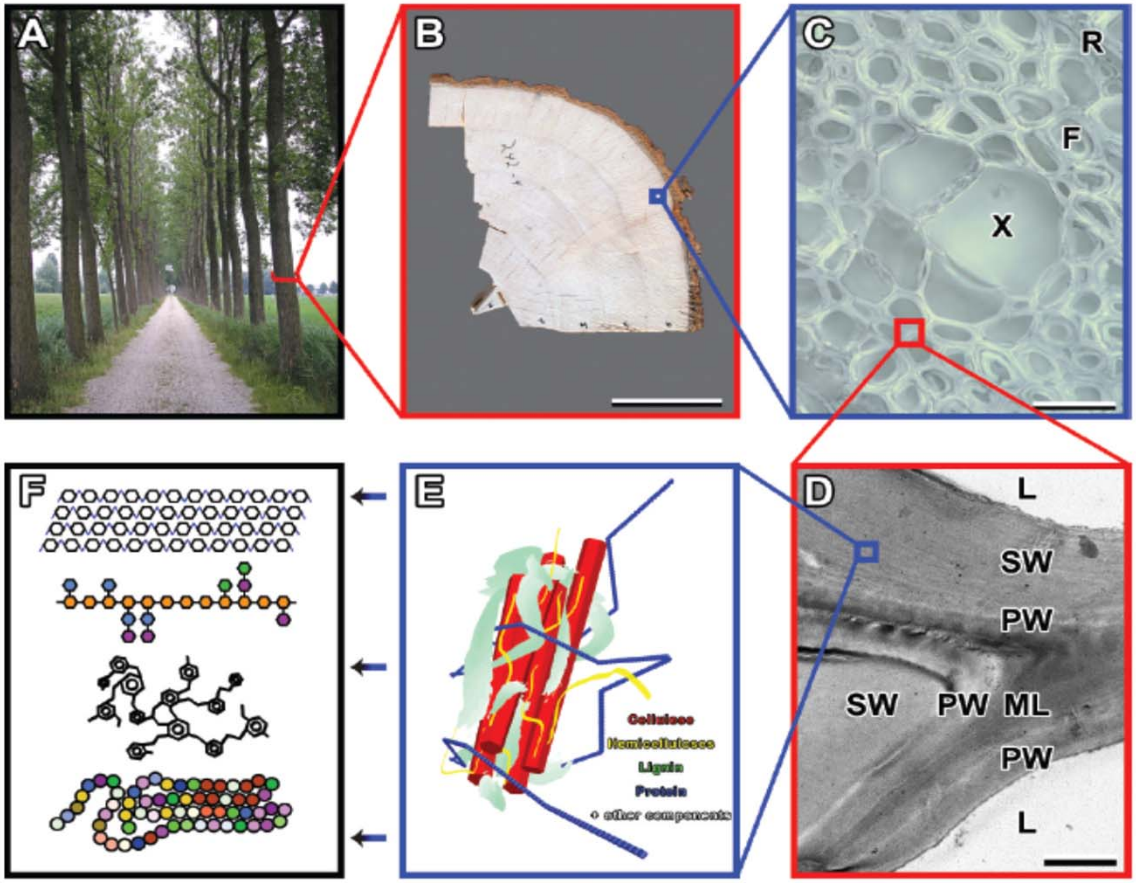
Induced Precipitation

Micro-organism mediated

What are the bio-char structure associated to each of these mechanisms? How can we enhance the formation of carbonaceous structures relevant for targeted pollutant removal?

Introduction

Scale down through biomass from the organismal to the molecular level



(A) populus sp **(B)** poplar wood **(C)** cross section of a poplar sample. Cell types: X- xylem element, F: wood fiber R: ray parenchyma **(D)** transmission electron microphotograph of a poplar xylem **(E)** artistic representation of the plant cell wall macromolecular structure, red, cellulose microfibrils, yellow hemicellulose and pectins green: lignin, blue structural proteins. **(F)** artistic representation of plant cell wall polymers (from top) cellulose hemicelluloses, lignin and protein

Source: Haas T.J., Nimlos M.R., Donohoe B.S.: Real-time and post-reaction microscopic structural analysis of biomass undergoing pyrolysis. Energy & fuels 2009, 23, 3810-3817.

Introduction

The **cell wall** is built up by several layers:

(1) Middle Lamella **ML**

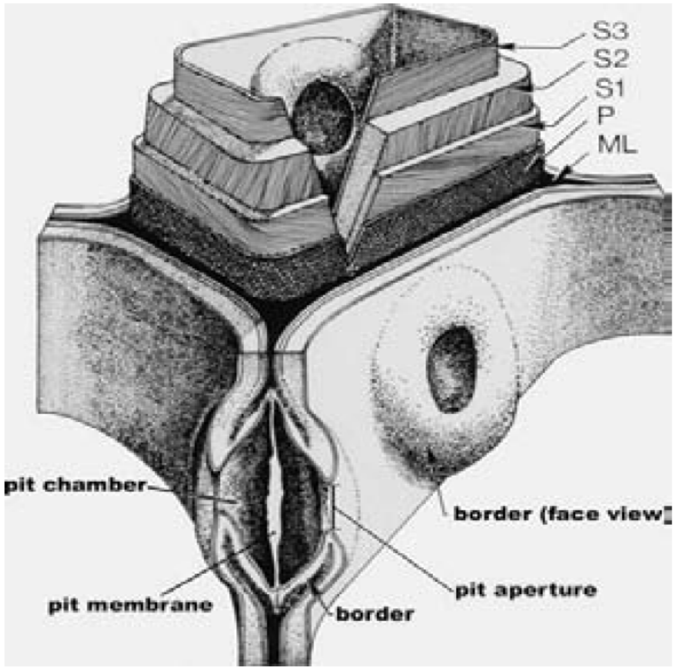
Binds the cells together

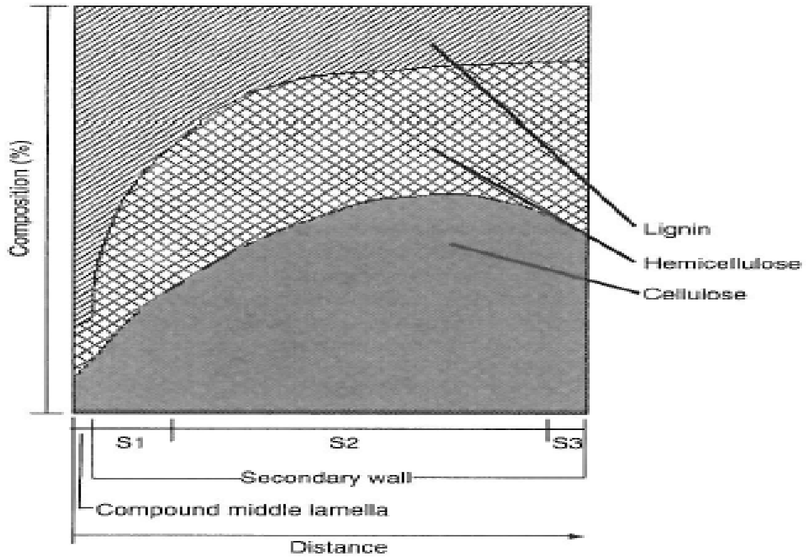
(2) Primary Wall (**P**)

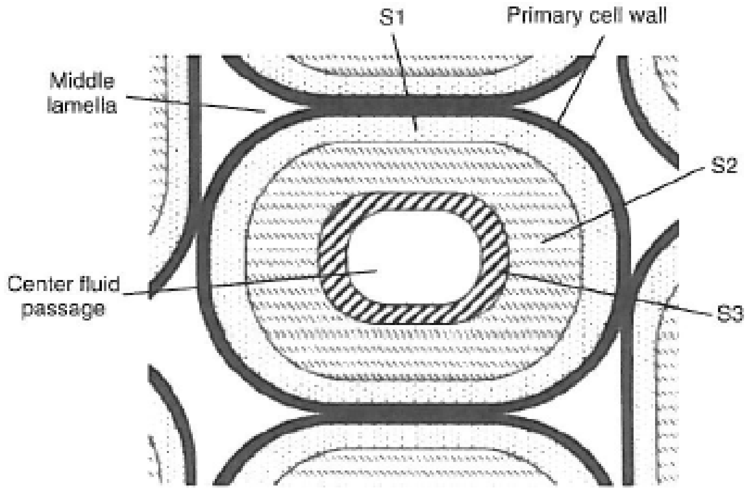
(3) Outer Layer of the Secondary Wall (**S_1**)

(4) Middle Layer of the Secondary Wall (**S_2**)

(5) Inner Layer of the Secondary Wall (**S_3**)







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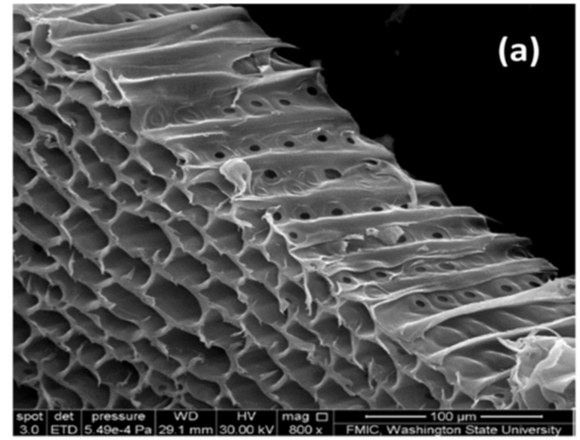
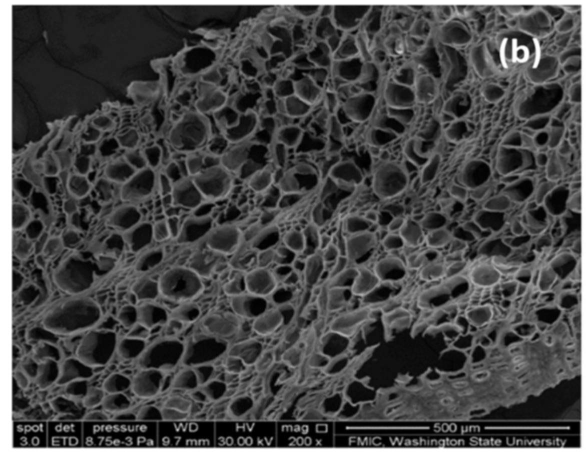
Introduction

Biochar conserves the structure of the Lignocellulosic Material!

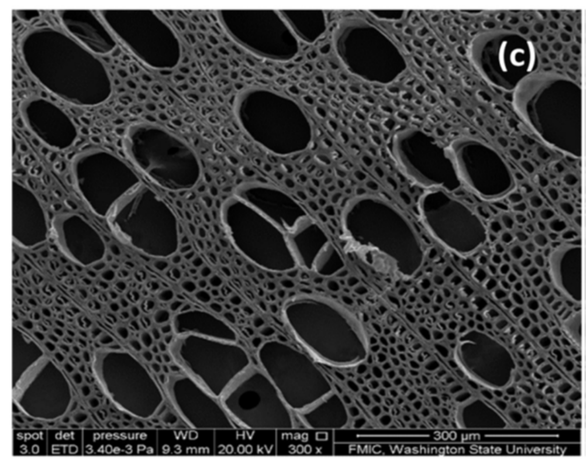
Douglas Fir Wood Char



Douglas Fir Bark Char

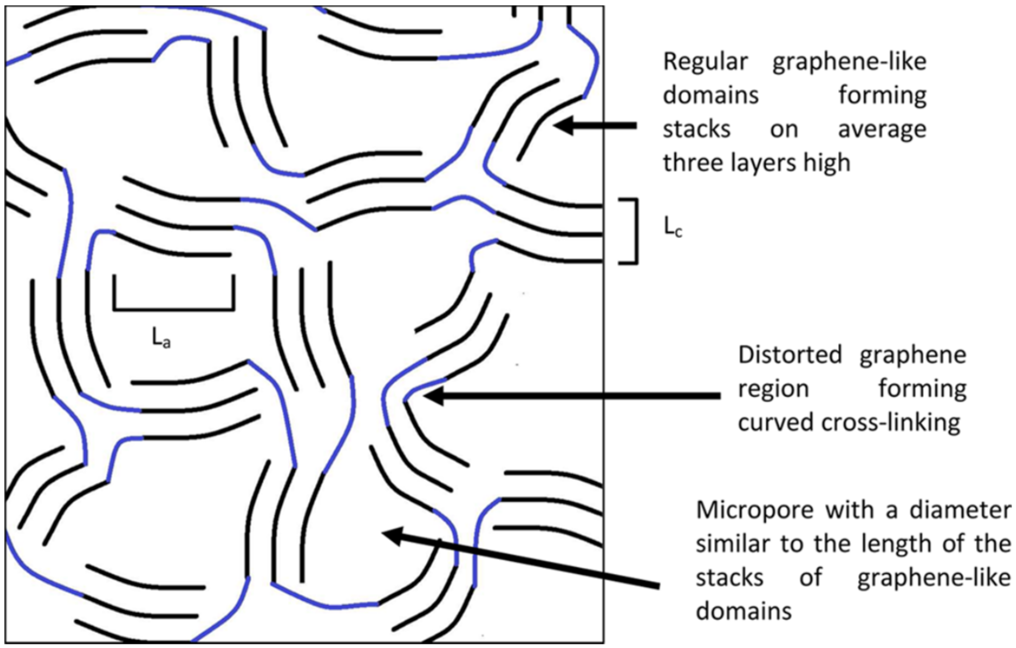


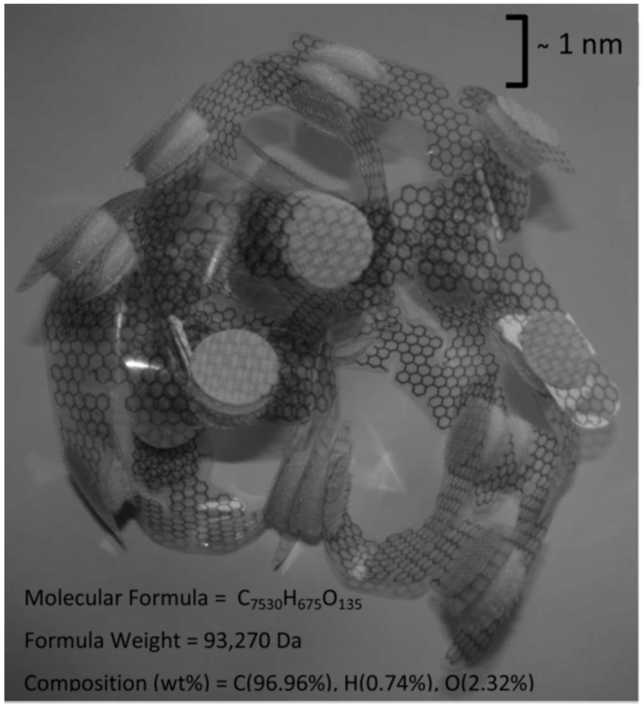
Hybrid Poplar



Introduction

McDonald-Wharry et al. 2016





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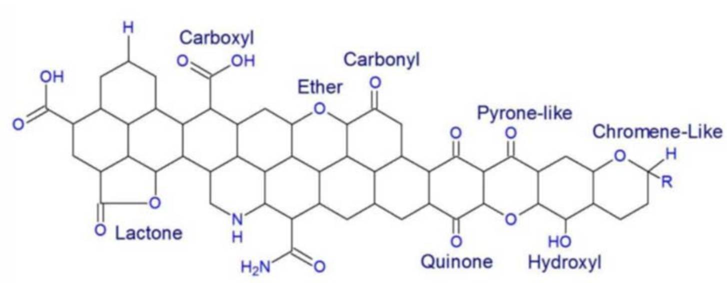
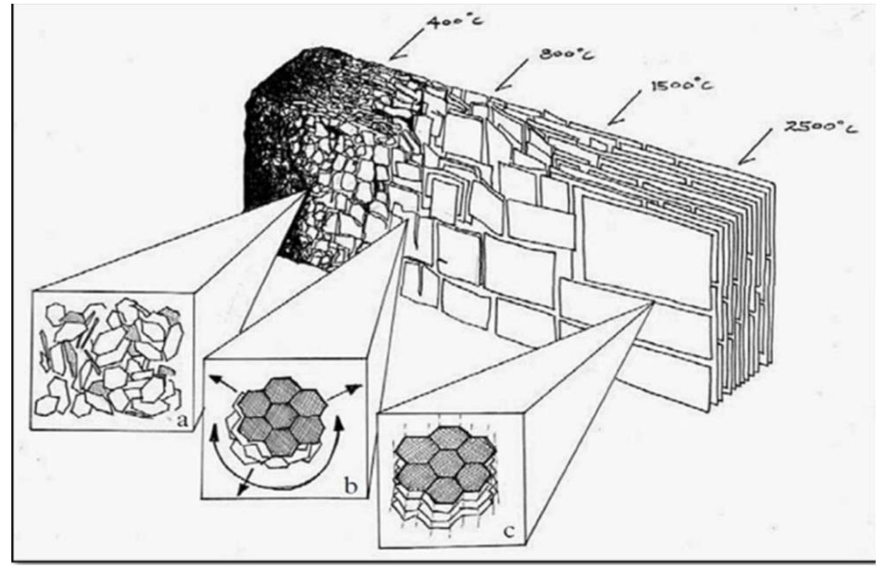
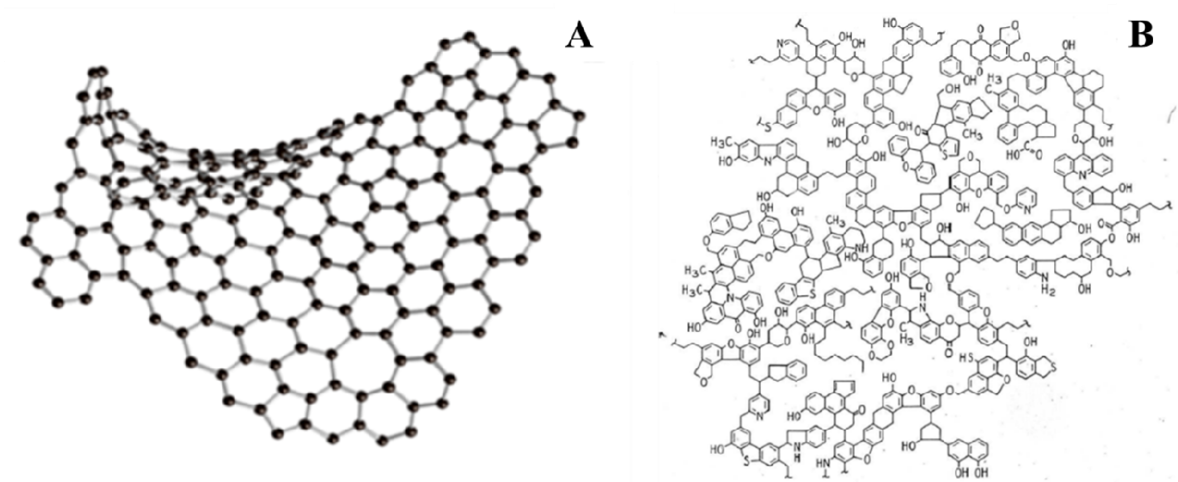
Introduction







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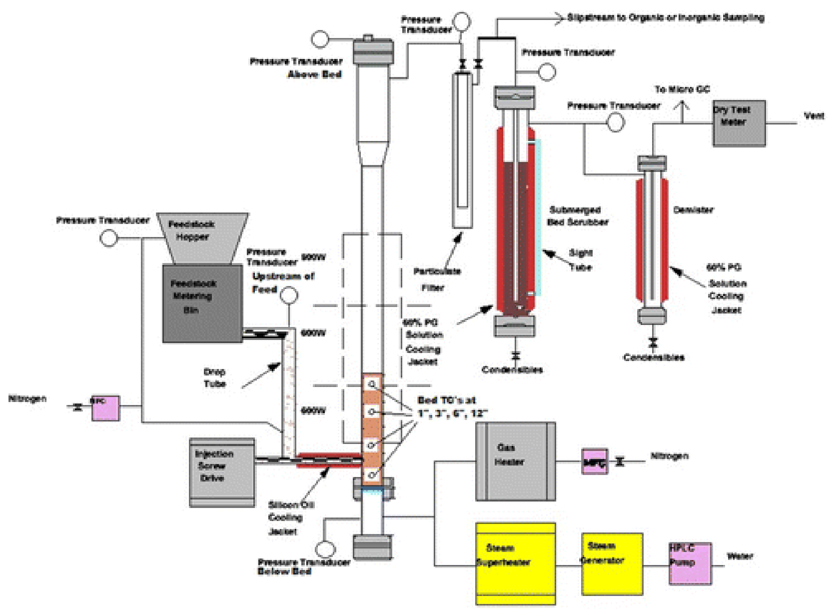
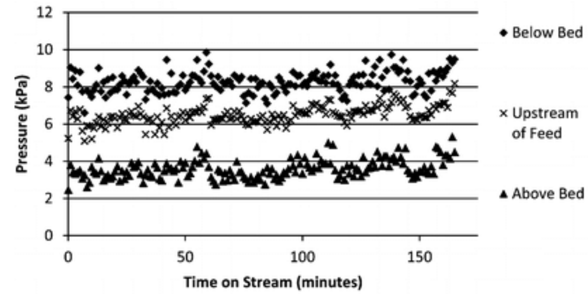
Formation of liquid intermediates (fundamentals)

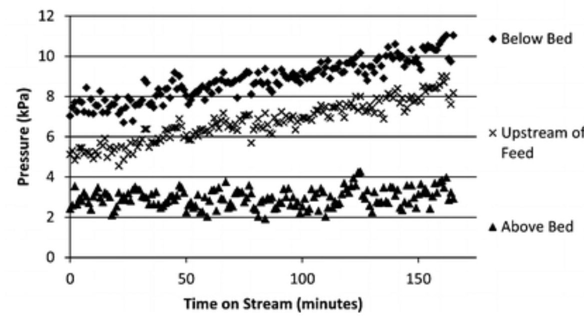
Gasification System

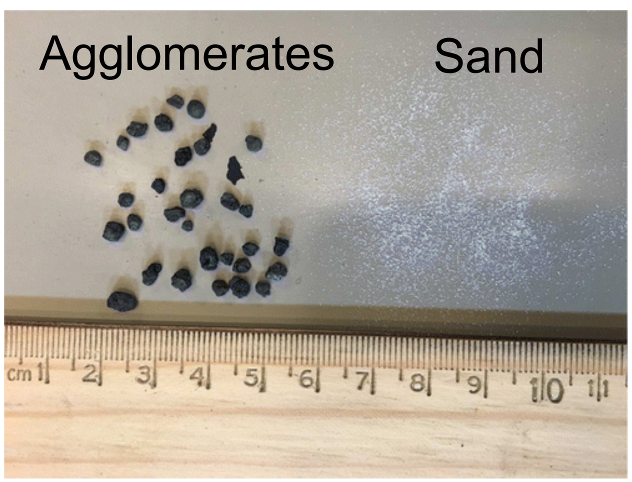


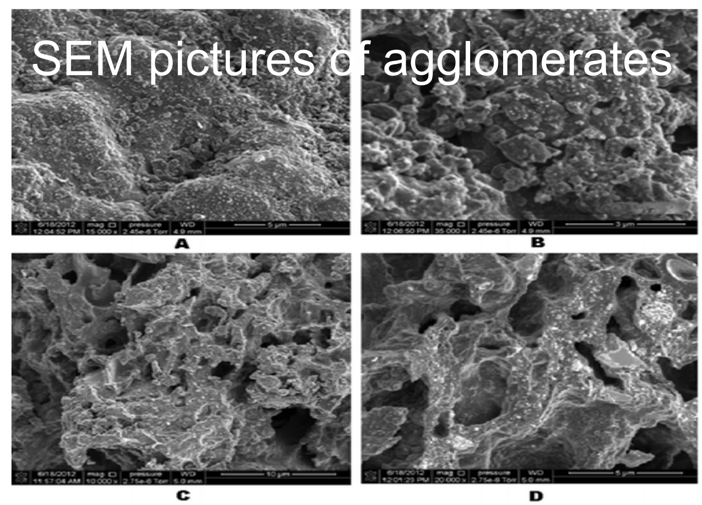
Corn Stover



Lignin rich residues







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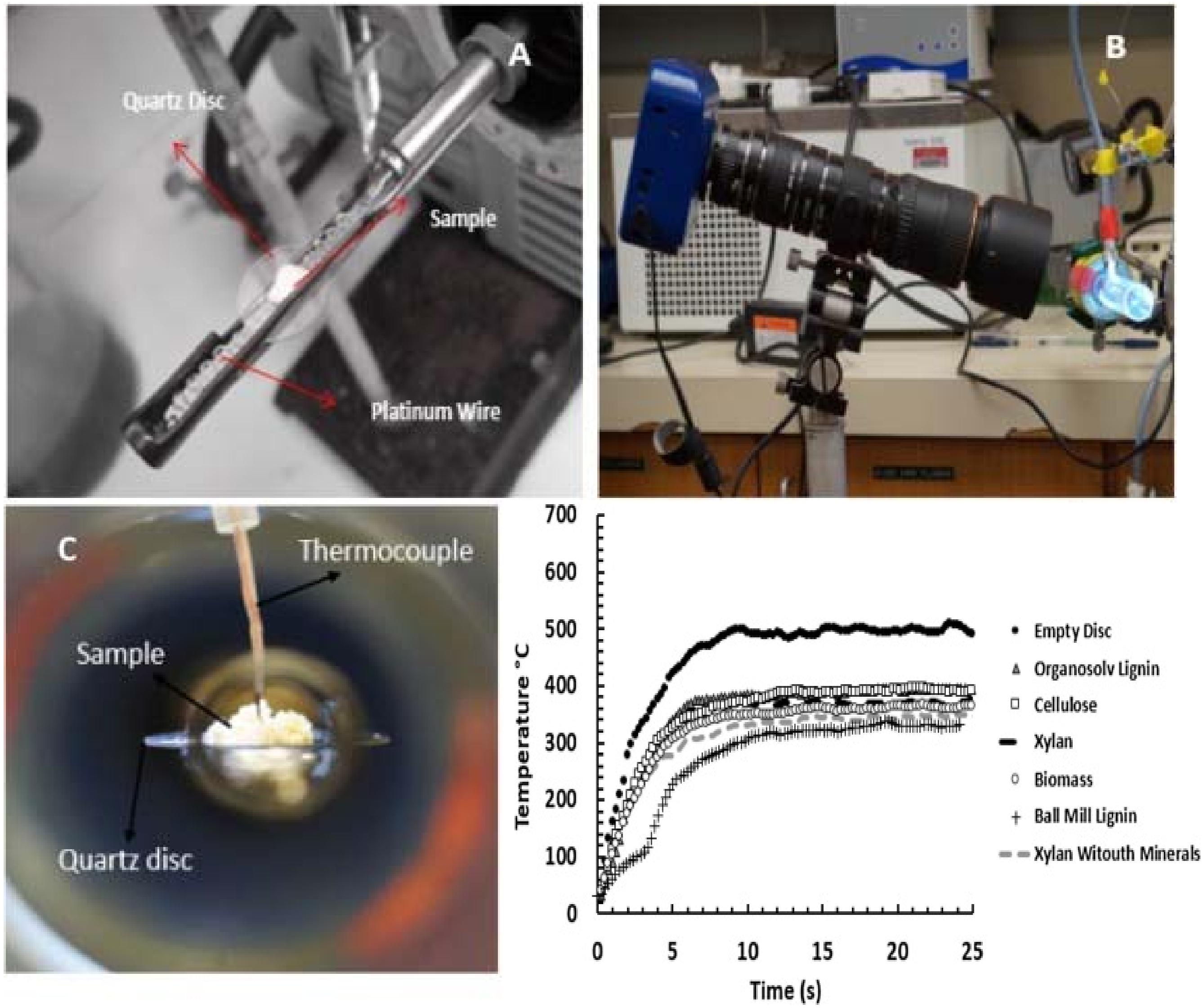
Formation of liquid intermediates (Fundamentals)



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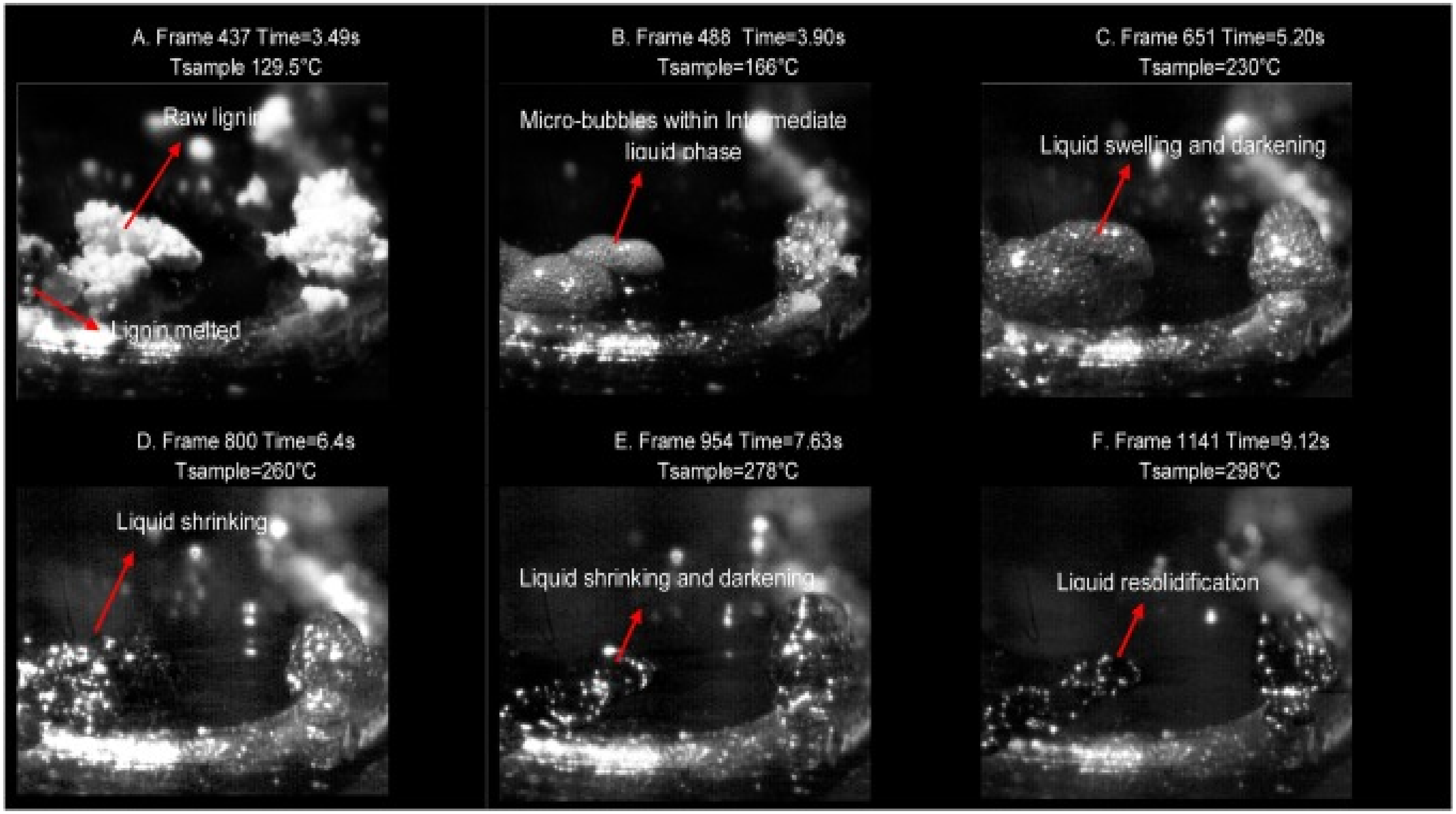
Formation of liquid intermediates

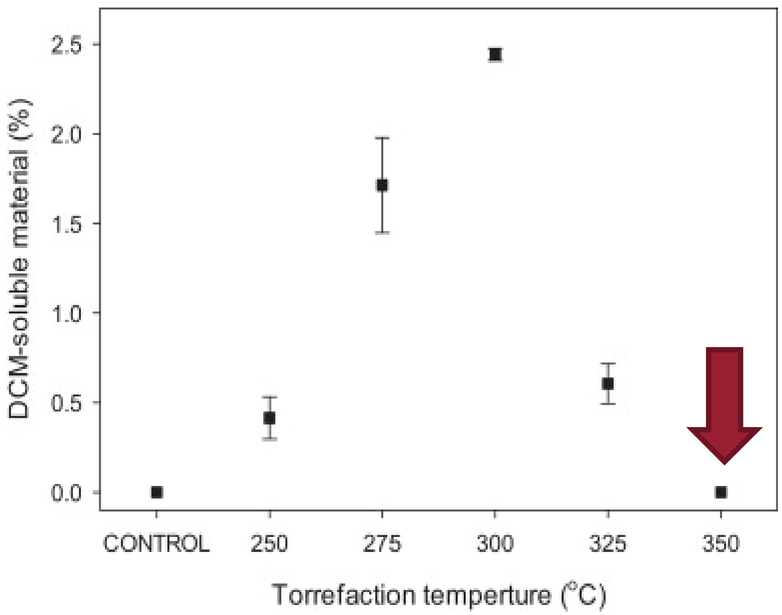
Formation of liquid Intermediates from Milled wood lignin



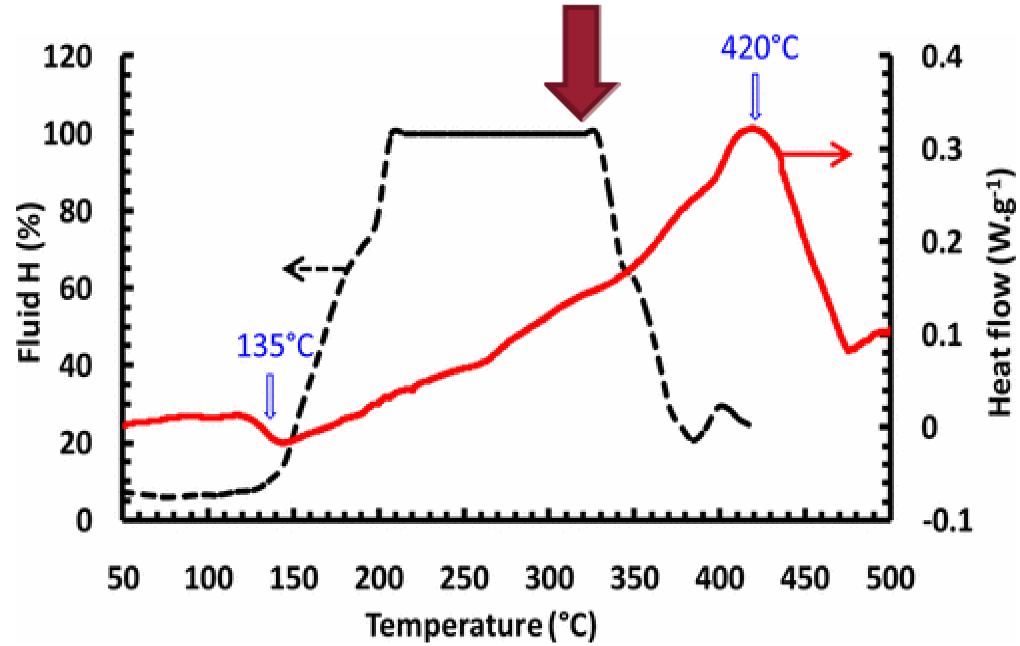
Montoya J, Pecha B, Chejne-Janna F, Garcia-Perez M: Identification of the fractions responsible for morphology conservation in lignocellulosic pyrolysis: Visualization studies of sugarcane bagasse and its pseudo-components. Journal of analytical and applied pyrolysis. 2017, 123, 307-318

Formation of liquid intermediates

Low molecular weight Oligomers (extractable with DCM) (Torrefaction of Ponderosa pine)



Fluidity development from in situ ^{1}H NMR and DSC (From Dr. Doufor's group)



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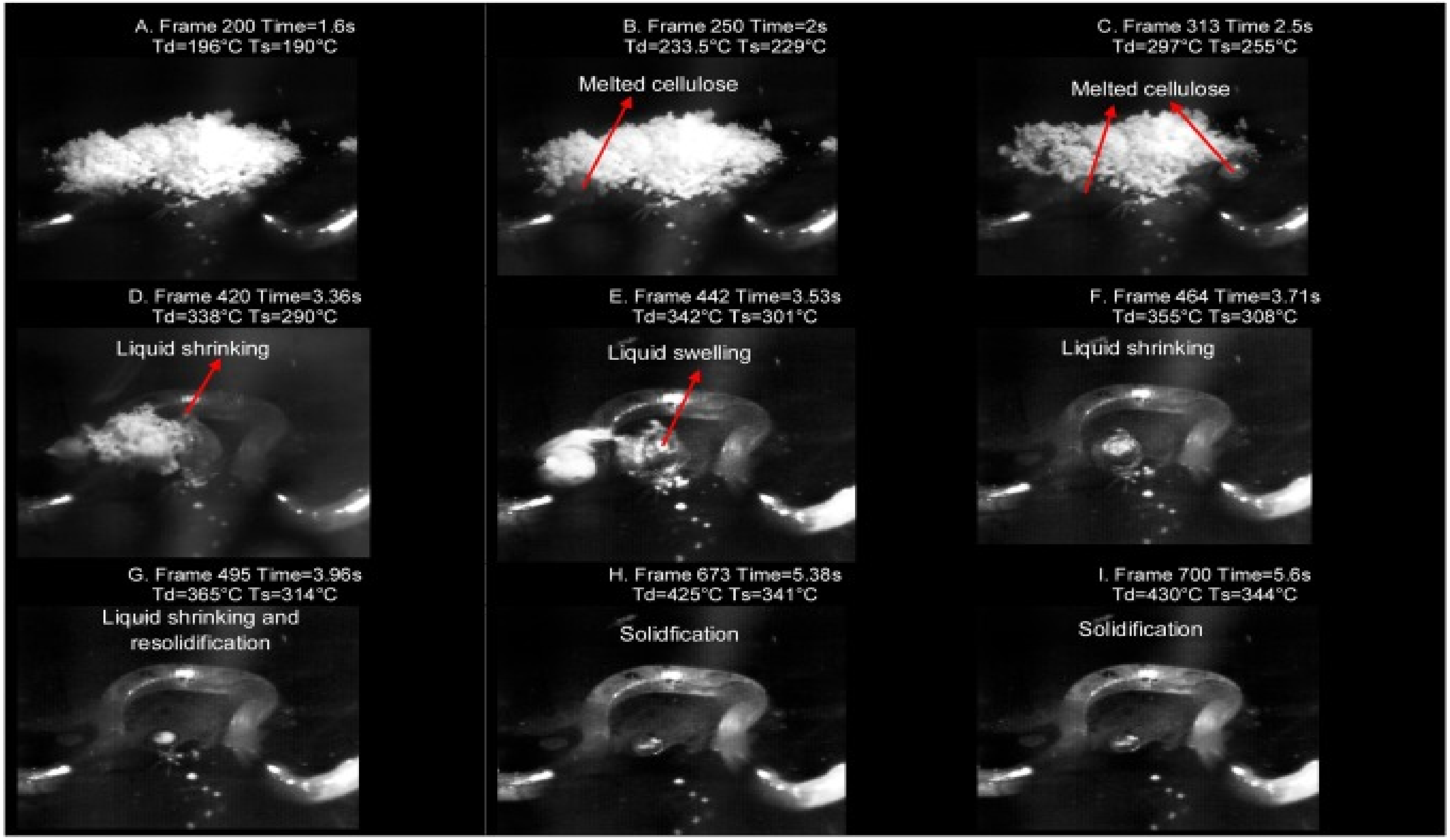
Formation of liquid intermediates

Cellulose melting and bubbling during Pyrolysis



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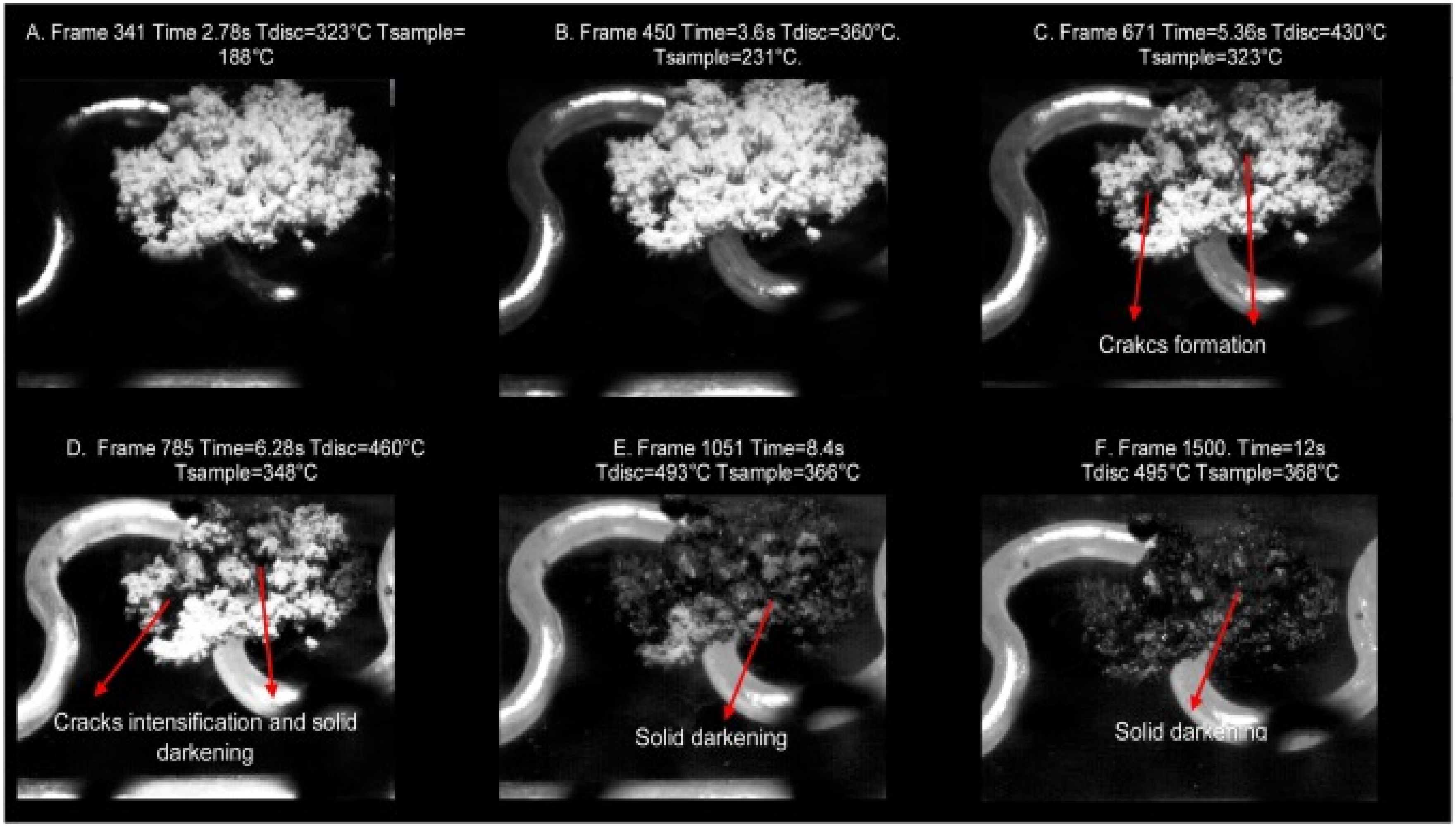
Formation of liquid intermediates

Xylan behavior during Pyrolysis



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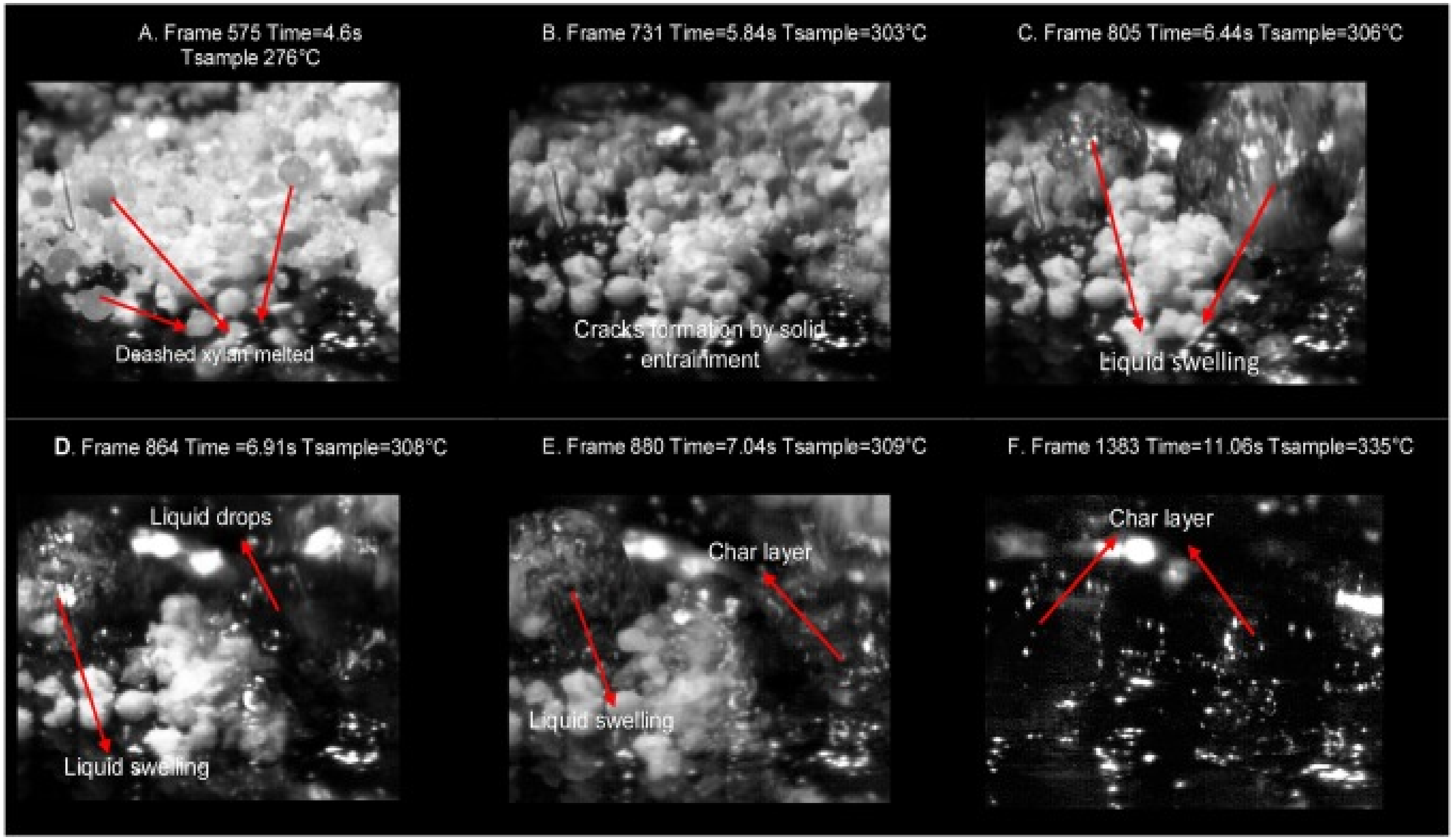
Formation of liquid intermediates

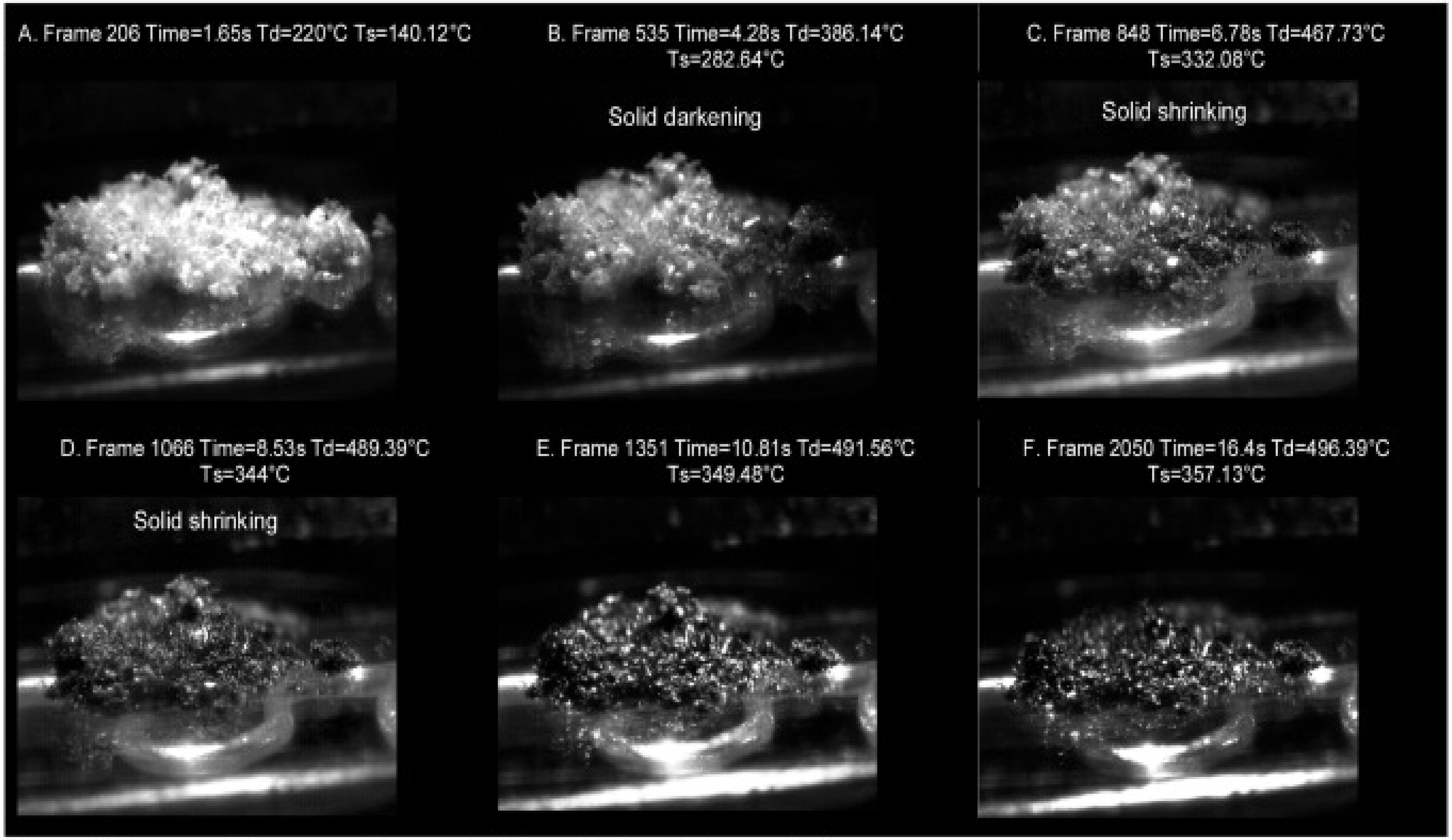
Deashed Xylan behavior during Pyrolysis



Montoya J, Pecha B, Chejne-Janna F, Garcia-Perez M: Identification of the fractions responsible for morphology conservation in lignocellulosic pyrolysis: Visualization studies of sugarcane bagasse and its pseudo-components. Journal of analytical and applied pyrolysis. 2017, 123, 307-318

Formation of liquid intermediates

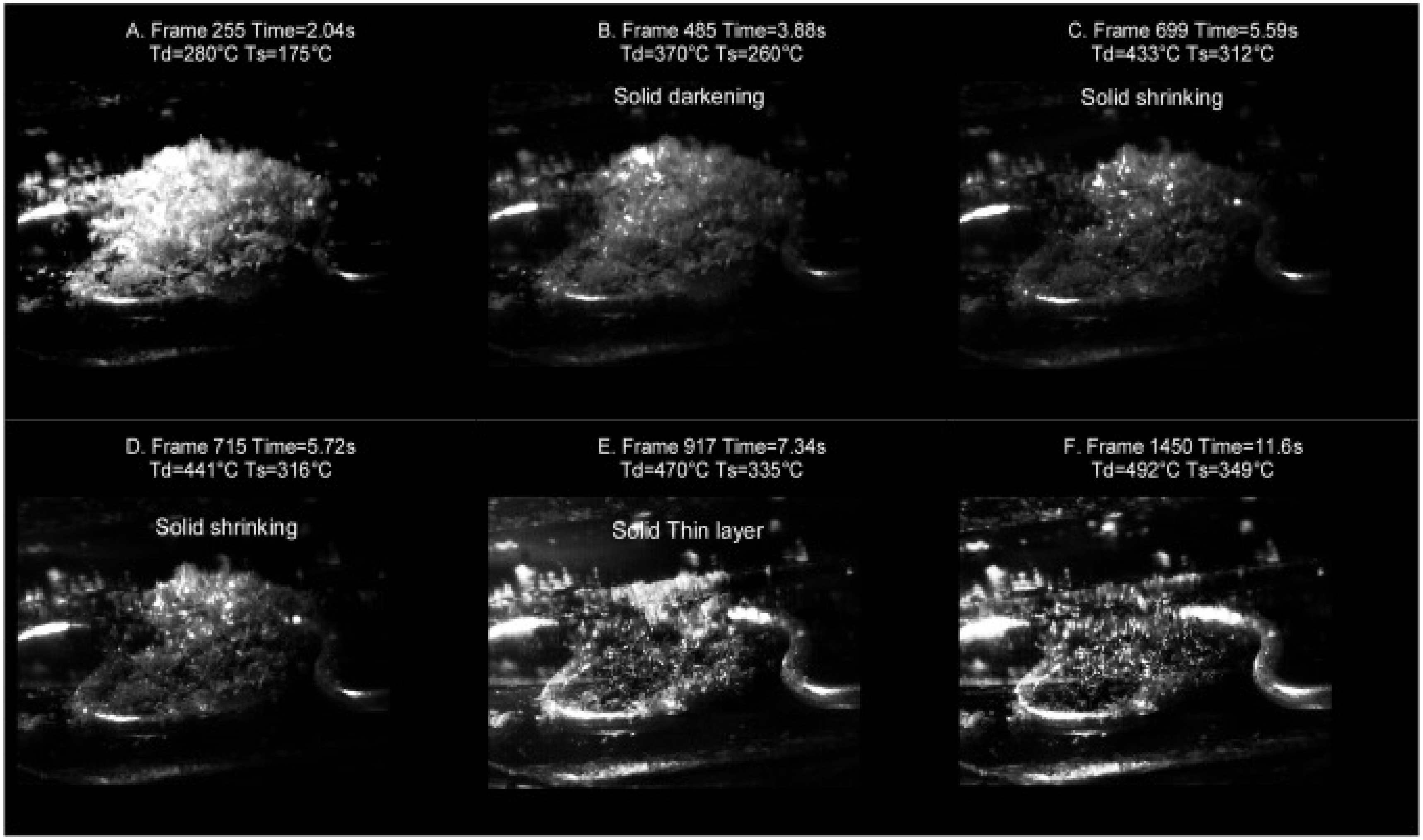
Sugarcane bagasse during Pyrolysis



Montoya J, Pecha B, Chejne-Janna F, Garcia-Perez M: Identification of the fractions responsible for morphology conservation in lignocellulosic pyrolysis: Visualization studies of sugarcane bagasse and its pseudo-components. Journal of analytical and applied pyrolysis. 2017, 123, 307-318

Formation of liquid intermediates

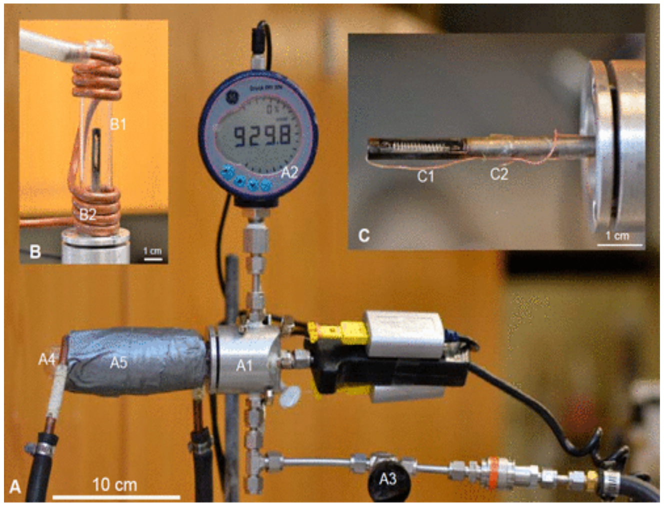
Acid washed sugar cane bagasse



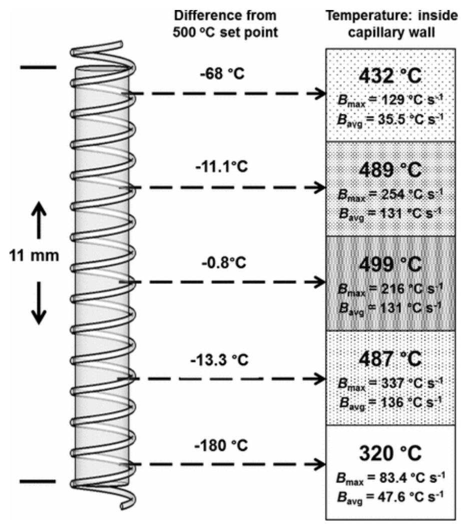
Montoya J, Pecha B, Chejne-Janna F, Garcia-Perez M: Identification of the fractions responsible for morphology conservation in lignocellulosic pyrolysis: Visualization studies of sugarcane bagasse and its pseudo-components. Journal of analytical and applied pyrolysis. 2017, 123, 307-318

Formation of liquid intermediates

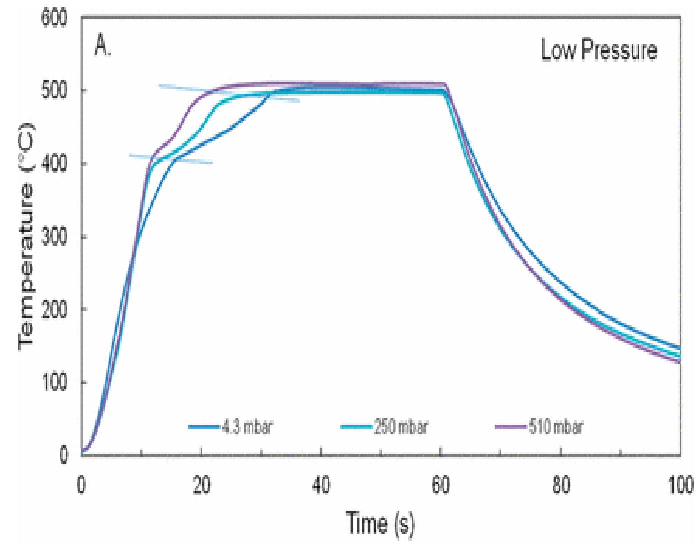
Modified Pyro-probe

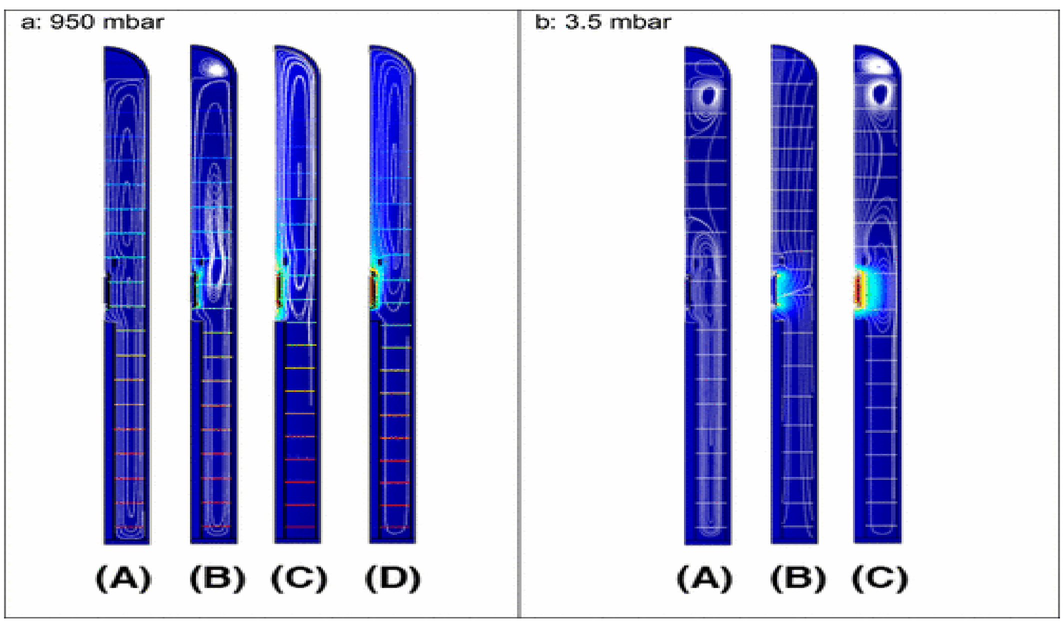


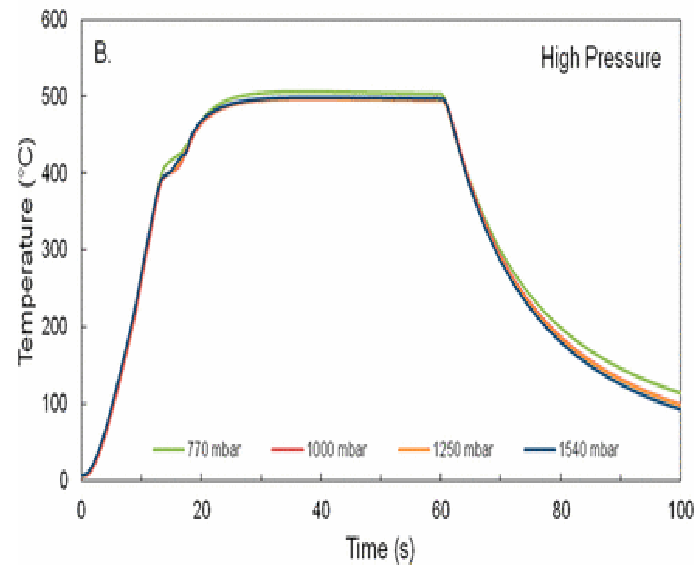
Temperature profile



Temperature profile



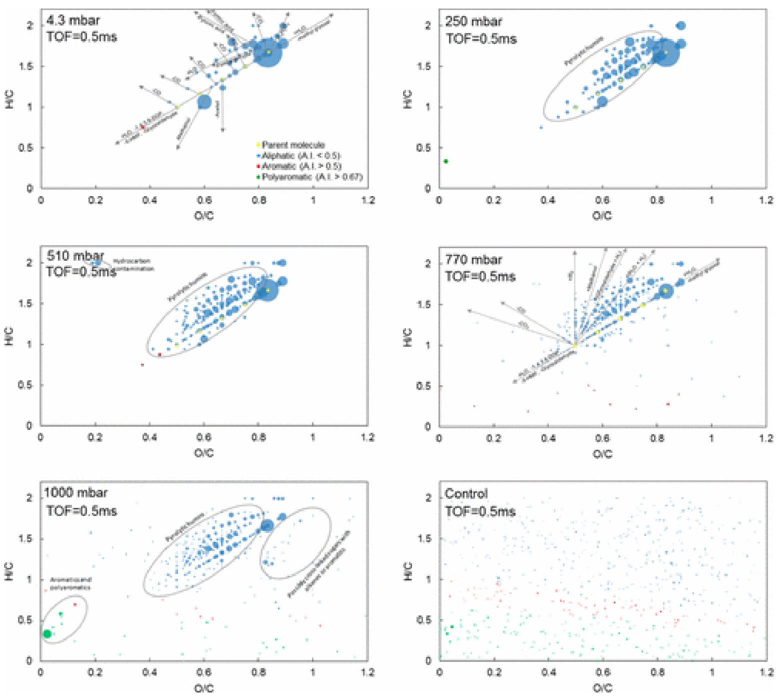




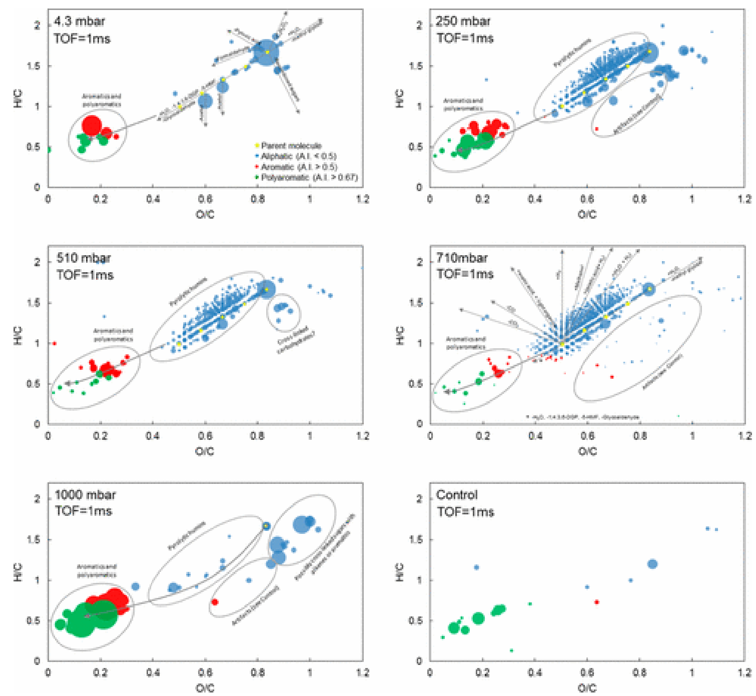
Formation of liquid intermediates

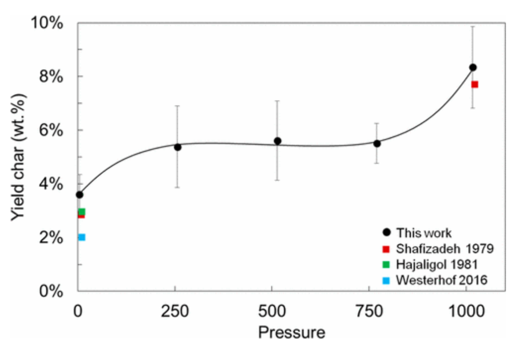
Reactions in the Liquid Intermediates (Cellulose) (FT-ICR-MS)

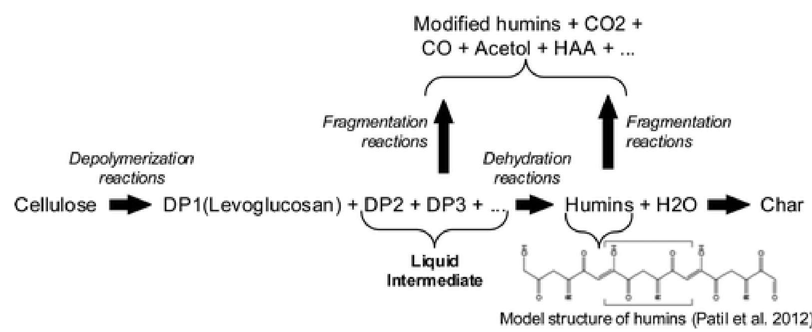
Small Oligomers



Large Oligomers







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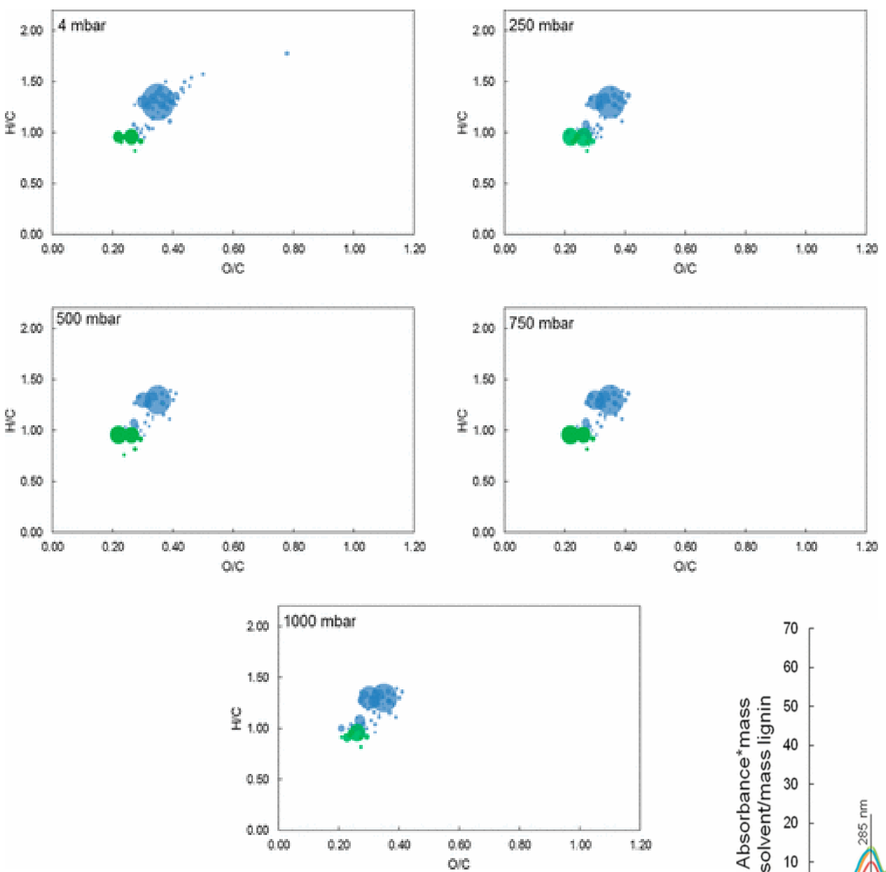
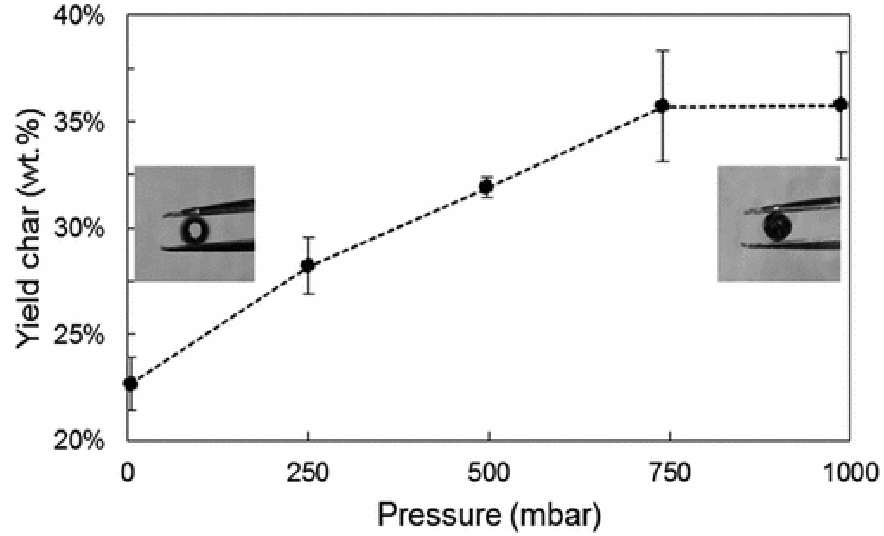
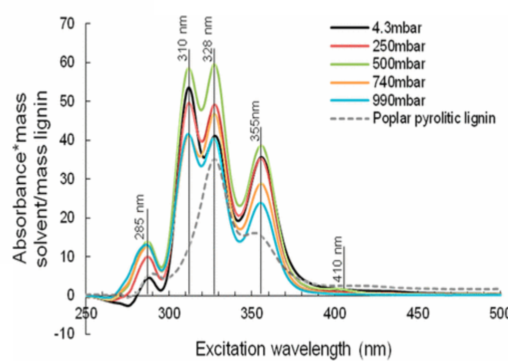
Formation of liquid intermediates

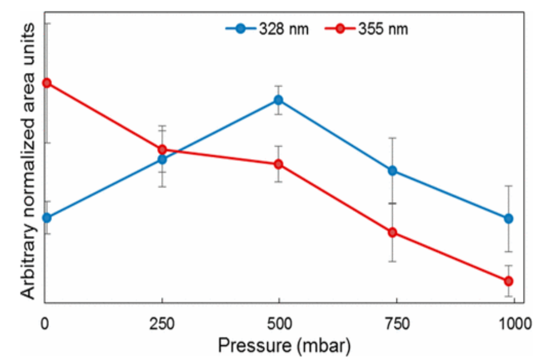
Reactions in the Liquid Intermediates (Lignin) (FT-ICR-MS)





UV-Fluorescence

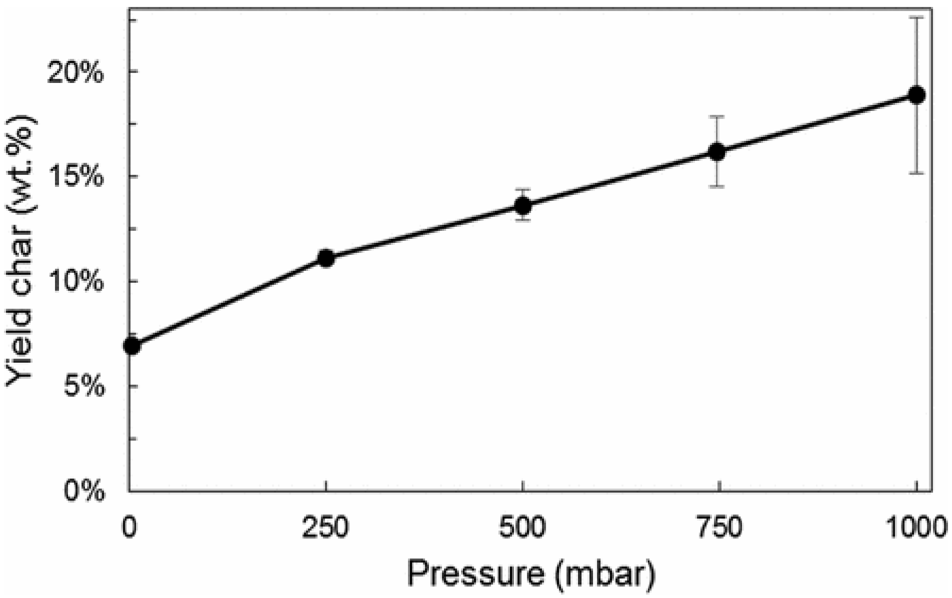


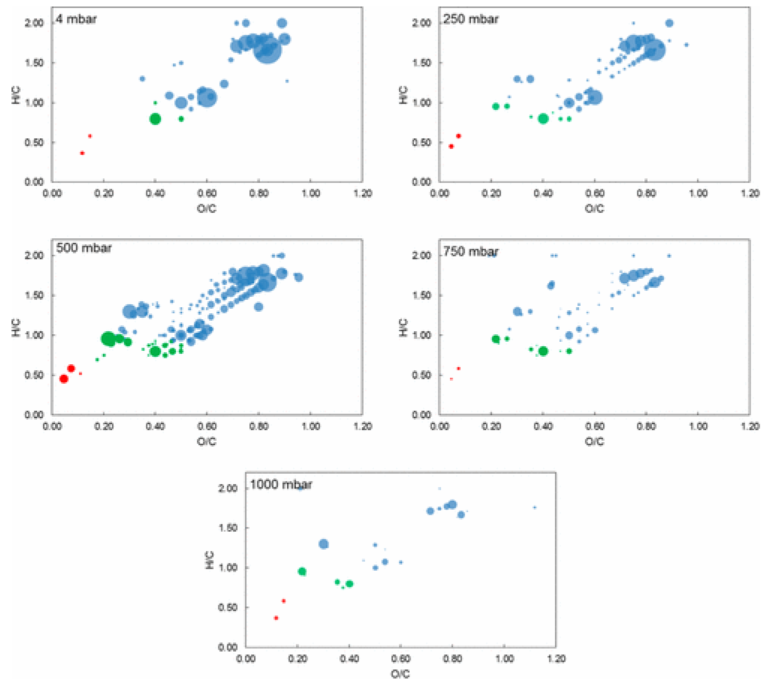


Pecha BM, Terrell E, Montoya JI, Stankovikj F, Broadbelt LJ, Chejne F, Garcia-Perez M: Effect of Pressure on Pyrolysis of Milled Wood Lignin and Acid-Washed Hybrid Poplar Wood. *Ind. Eng. Chem. Res.*, 2017, In press

Formation of liquid intermediates

Reactions in the Liquid Intermediates (Sugarcane Bagasse) (FT-ICR-MS)

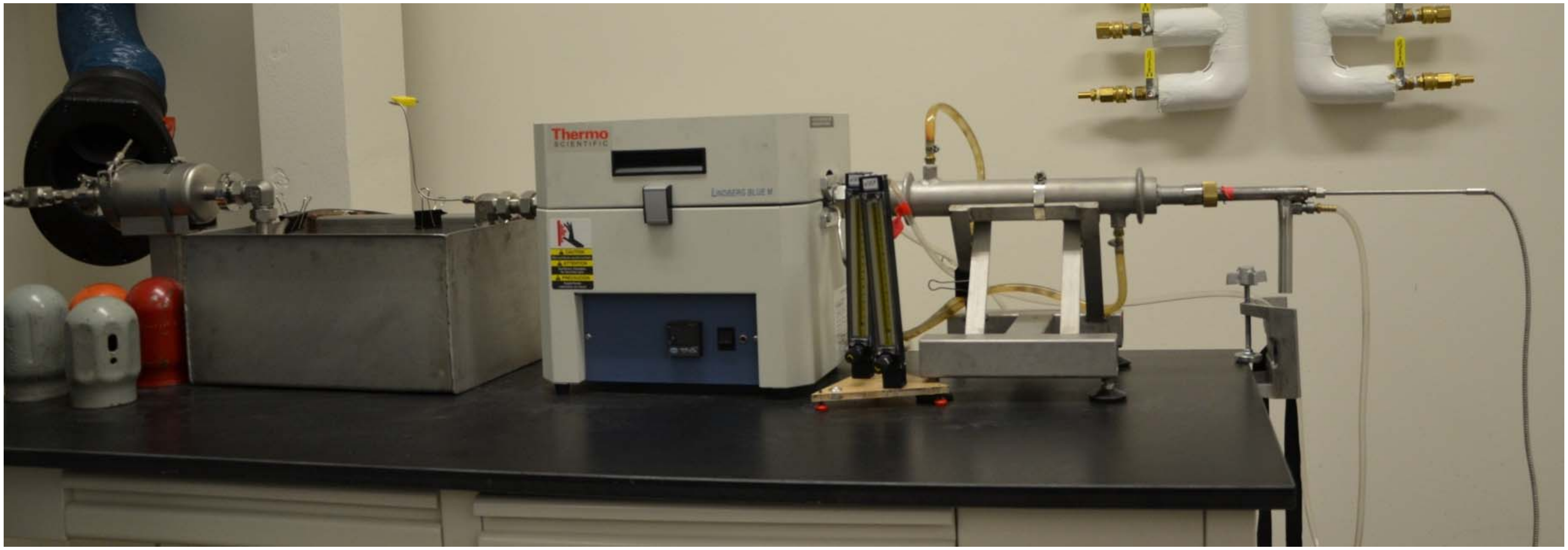


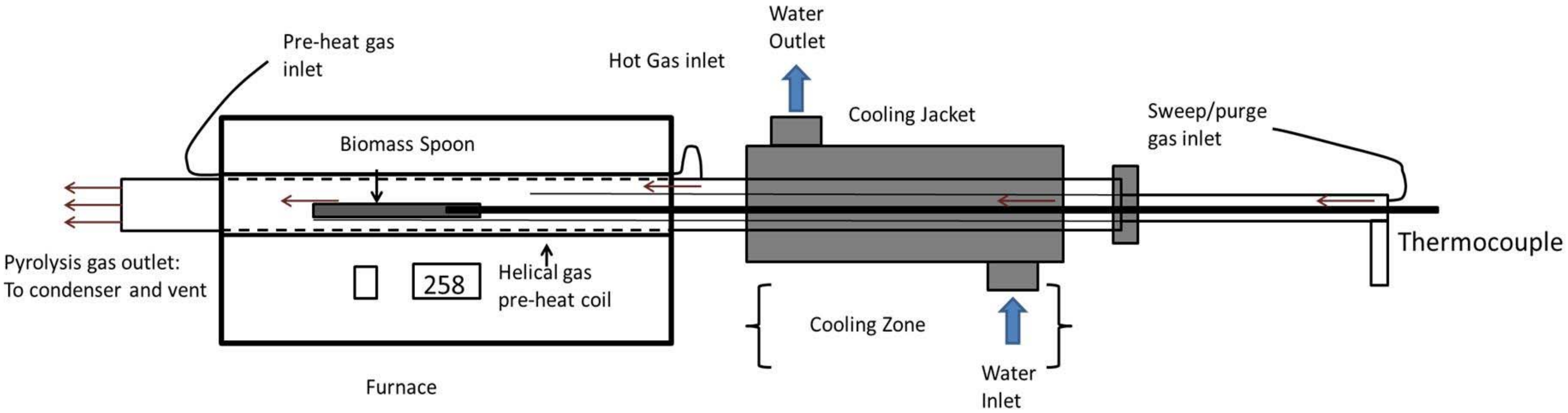


Pecha BM, Terrell E, Montoya JI, Stankovikj F, Broadbelt LJ, Chejne F, Garcia-Perez M: Effect of Pressure on Pyrolysis of Milled Wood Lignin and Acid-Washed Hybrid Poplar Wood. *Ind. Eng. Chem. Res.*, 2017, In press

Biochar Formation Mechanisms

Spoon Reactor

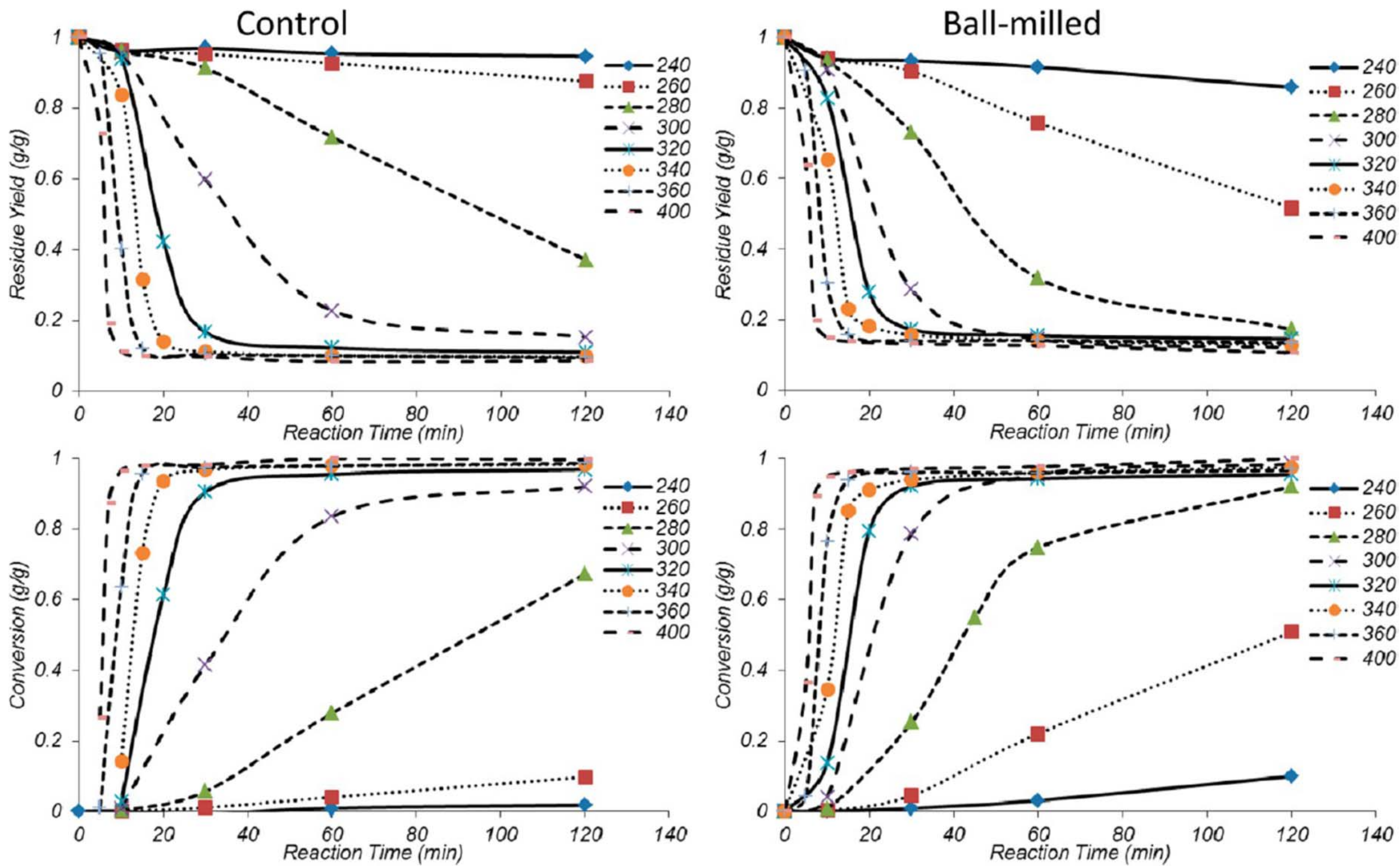




Wang Z, Pecha B, Westerhof RJM, Kersten SRA, Li C-Z, McDonald AG, Garcia-Perez M: Effect of Cellulose Crystallinity on Solid/Liquid Phase Reactions Responsible for the Formation of Carbonaceous Residues during Pyrolysis. Industrial & Engineering Chemistry Research, 2014, 53, 2940-2955

Biochar Formation Mechanisms

Study: Formation Mechanisms of Cellulose Char



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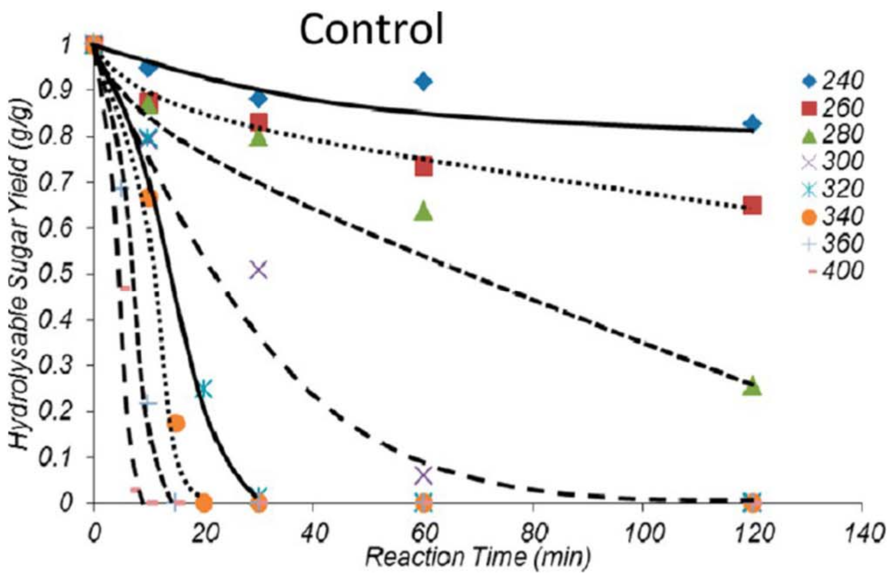
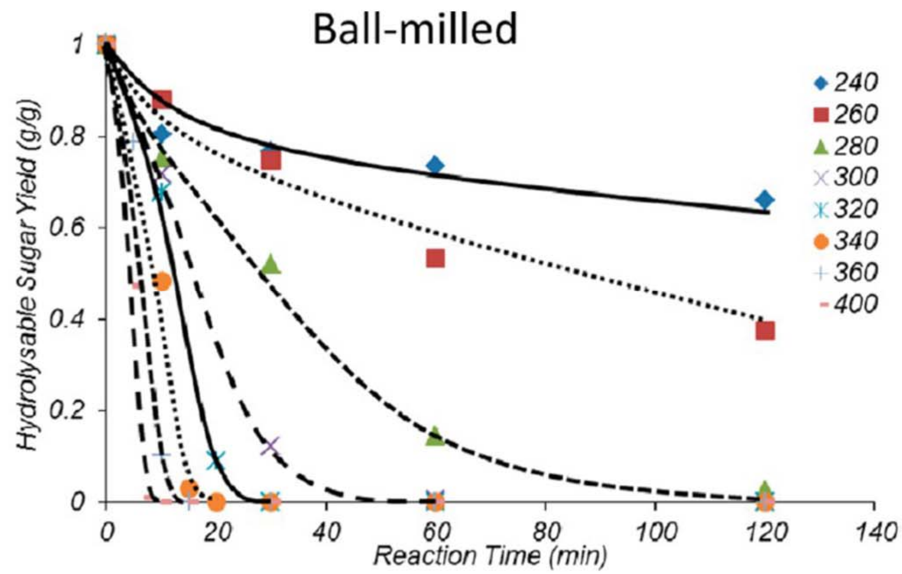
Biochar Formation Mechanisms

Content of hydrolysable sugar





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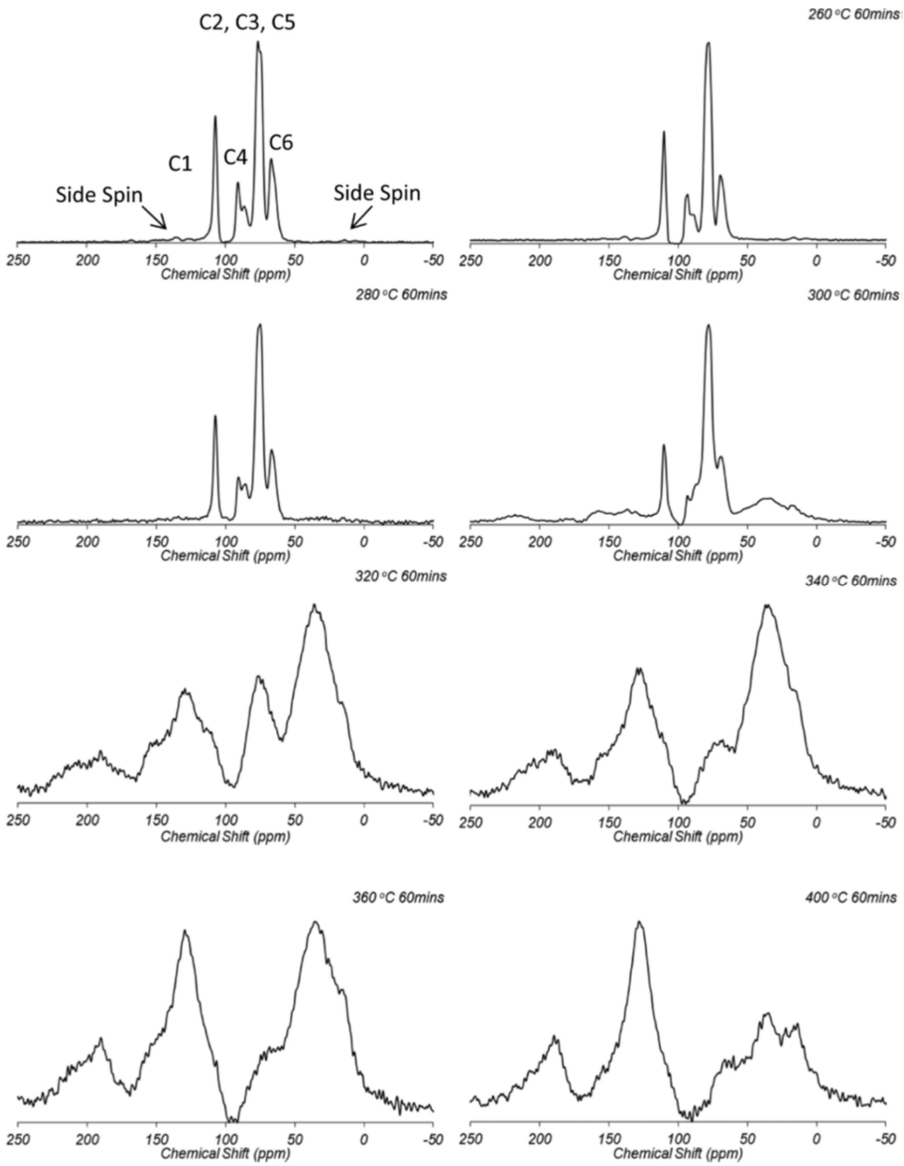
Biochar Formation Mechanisms



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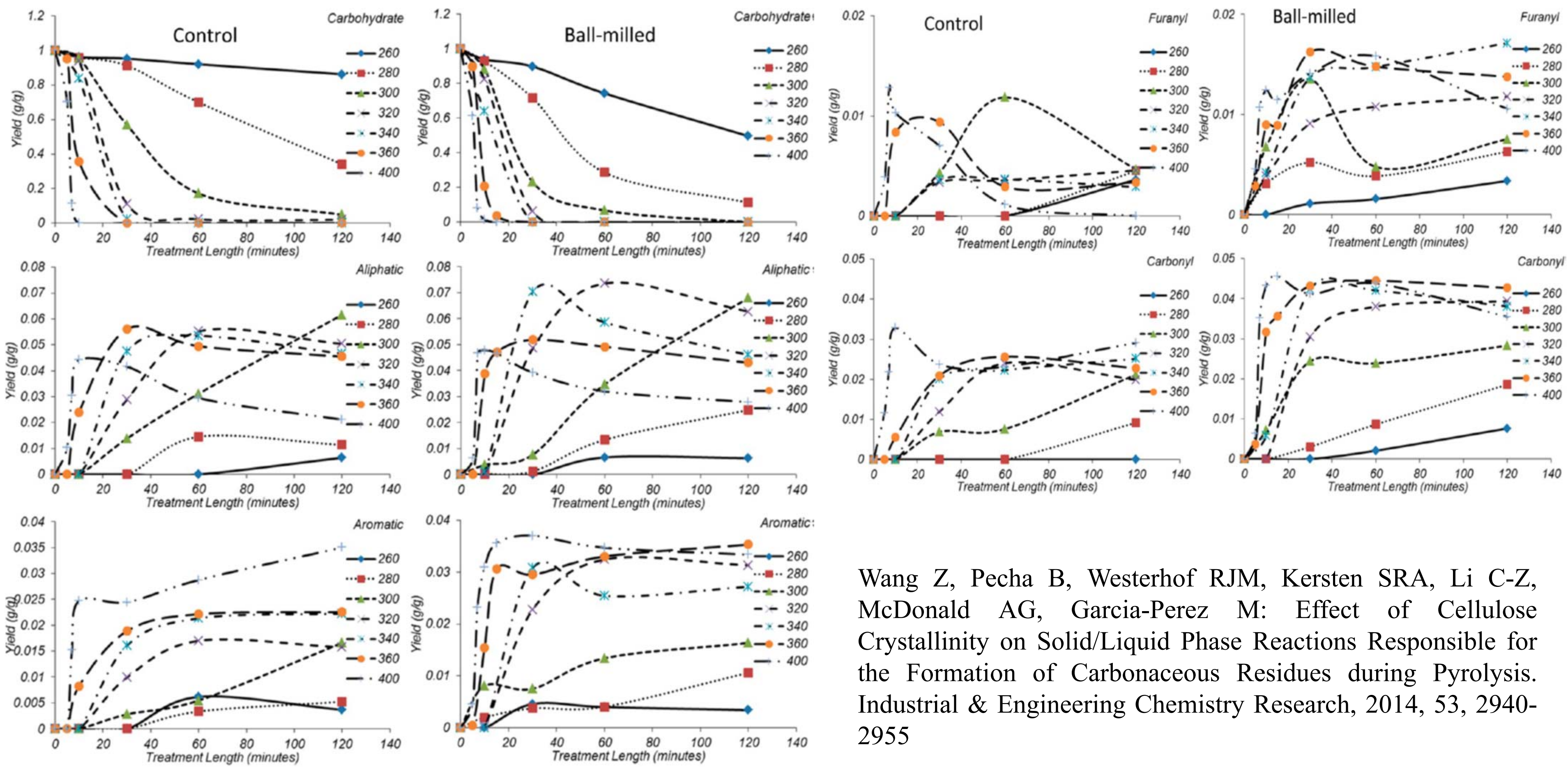
Biochar Formation Mechanisms

Yield of carbohydrate, aliphatic, aromatic, furanyl and carbonyl groups

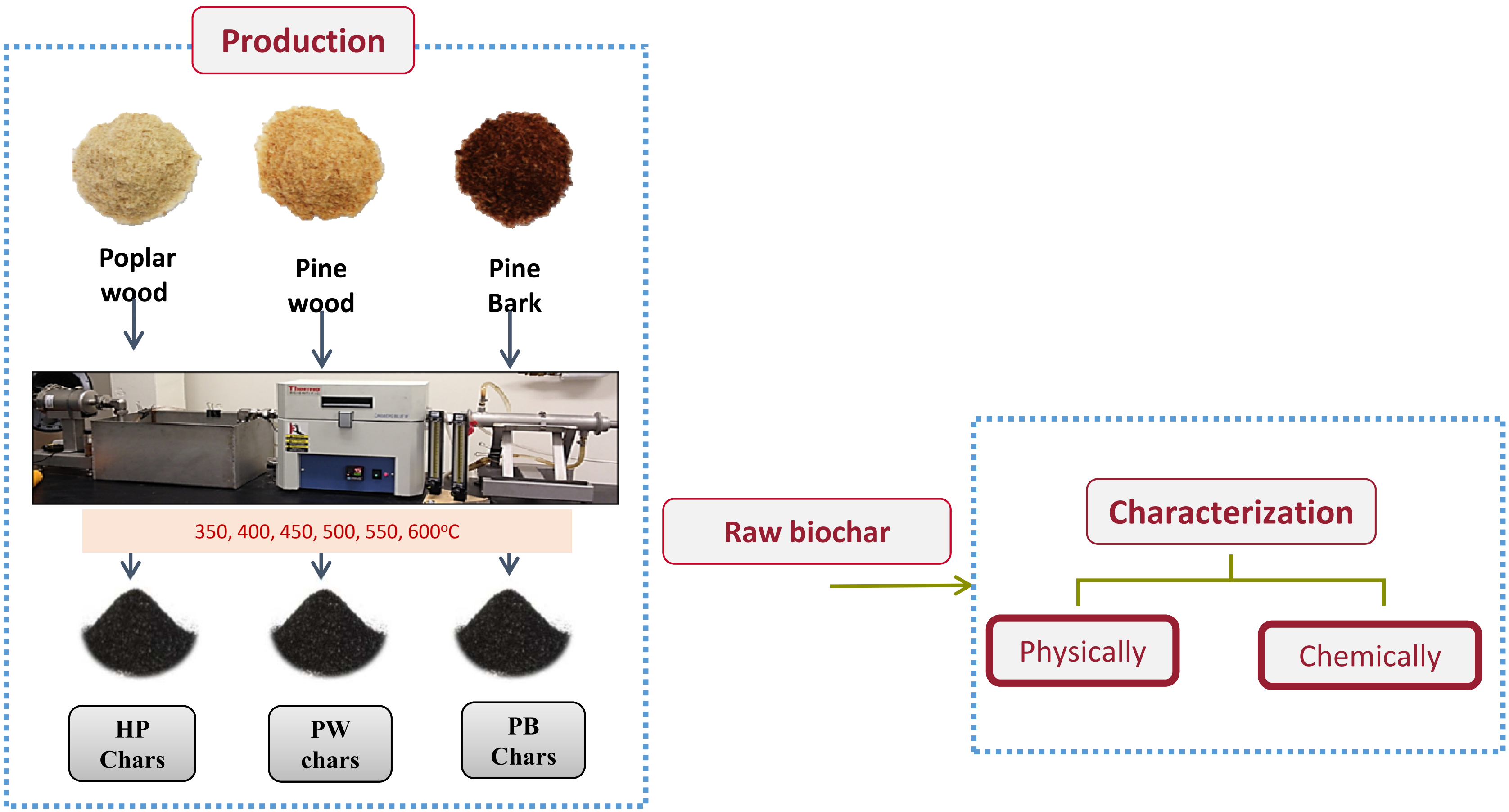


Wang Z, Pecha B, Westerhof RJM, Kersten SRA, Li C-Z, McDonald AG, Garcia-Perez M: Effect of Cellulose Crystallinity on Solid/Liquid Phase Reactions Responsible for the Formation of Carbonaceous Residues during Pyrolysis. Industrial & Engineering Chemistry Research, 2014, 53, 2940-2955

The relationship between biochar structure, composition and adsorption capacity is poorly known

Biochar Properties

Study: Effect of Pyrolysis Temperature



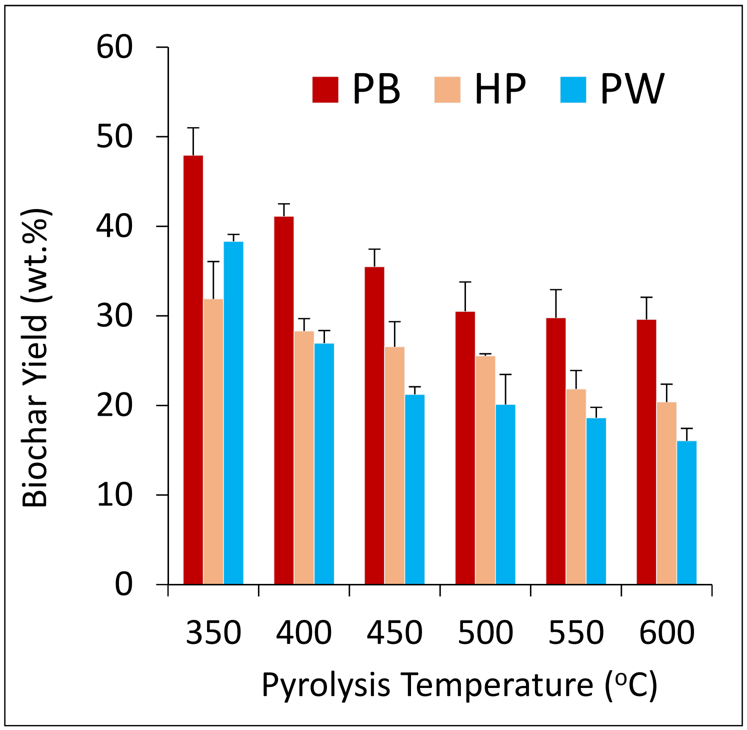
Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

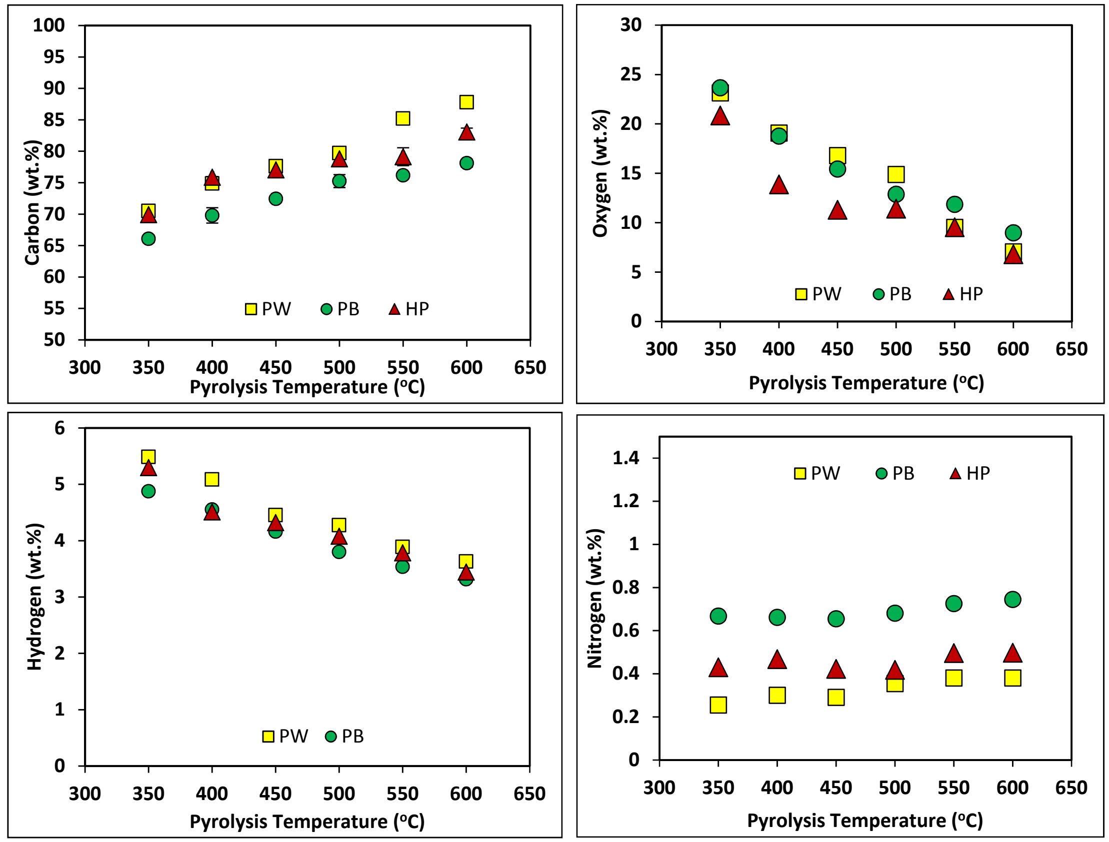
Biochar Properties

Effect of Pyrolysis Temperature

Elemental Composition

Yield of Char



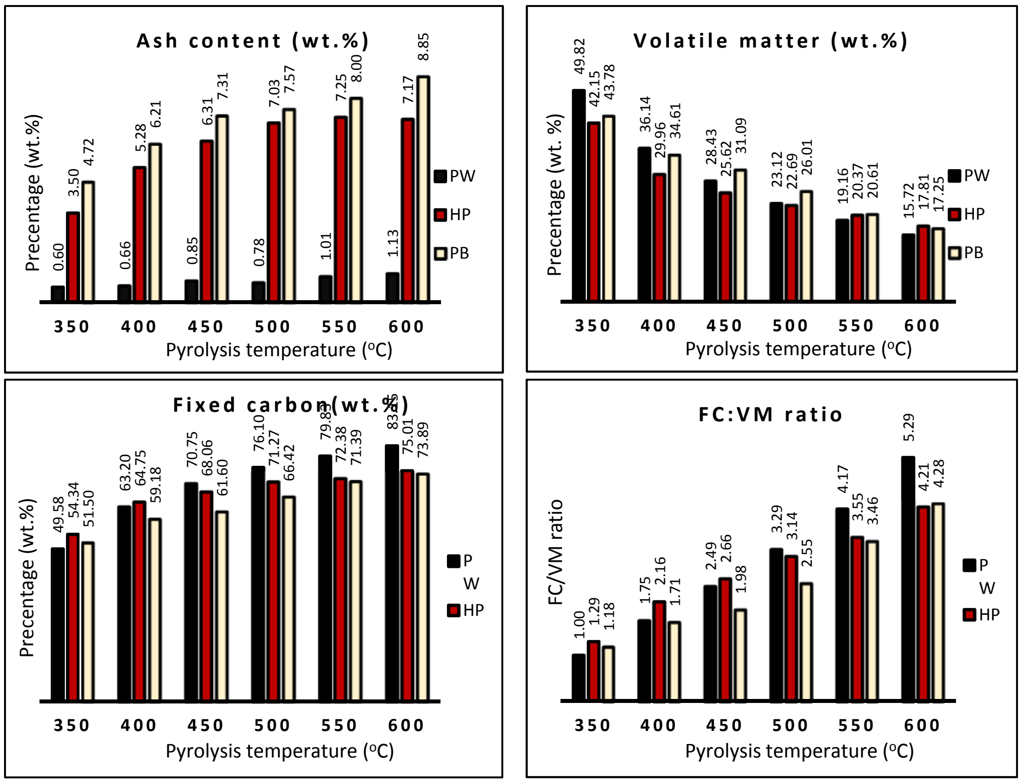


Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

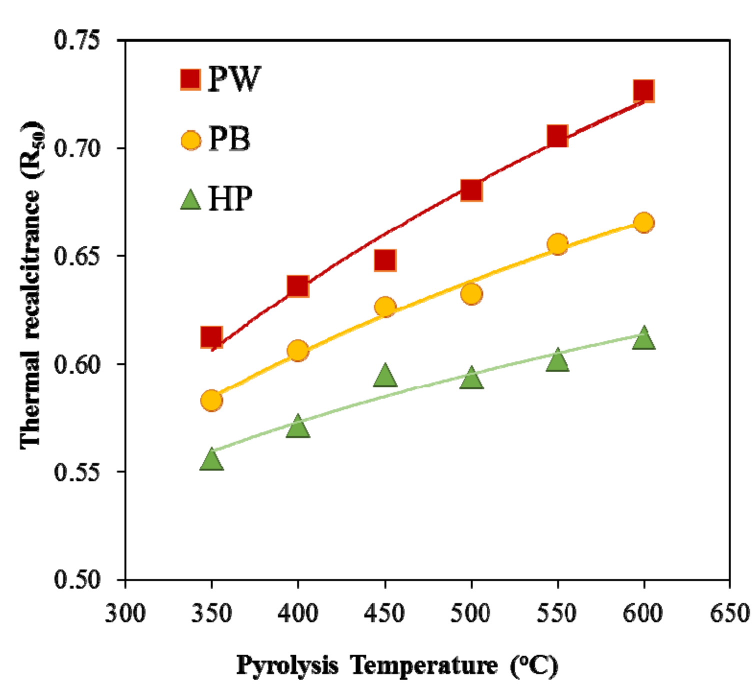
Biochar Properties

Effect of Pyrolysis Temperature

Proximate analysis



Thermal recalcitrance



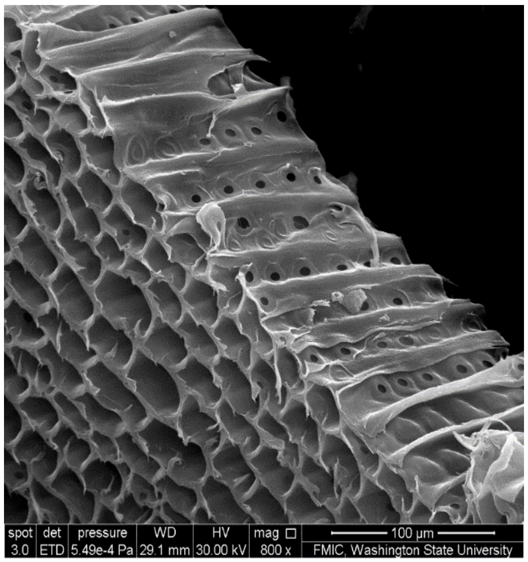
$$R_{50,\ biochar} = T_{50,\ biochar} / T_{50,\ graphite}$$

$T_{50,\ biochar}$ and $T_{50,\ graphite}$ were the temperature values corresponding to 50% weight loss by oxidation/volatilization of biochar and graphite

Suliman WSO, Harsh J, Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

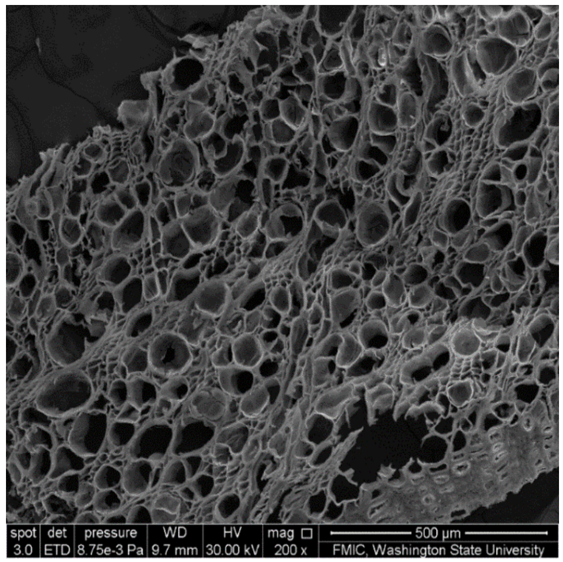
Biochar Properties

SEM Analysis



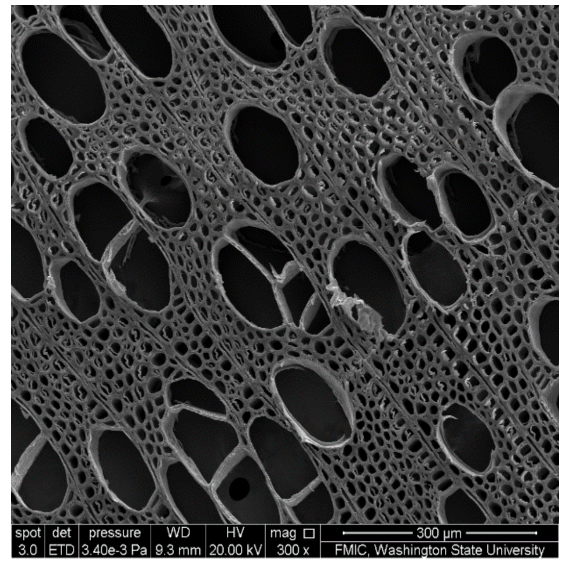
Pine wood biochar

mag. 800X



Pine bark biochar

mag. 200X



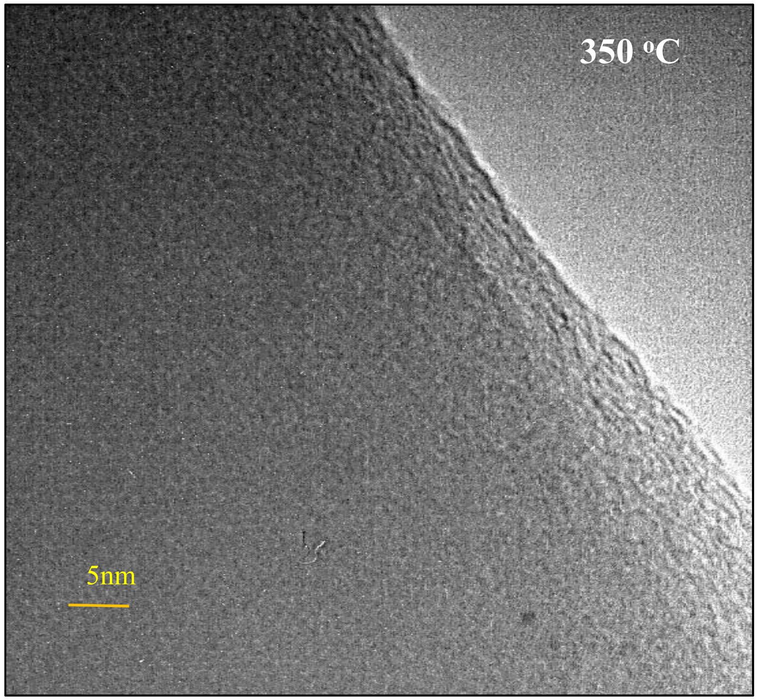
Poplar wood biochar

mag. 300X

Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

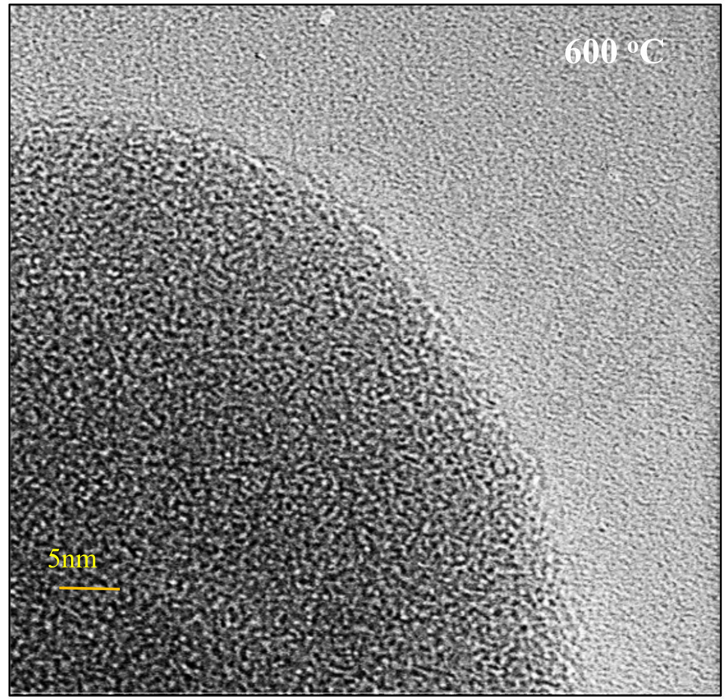
Biochar Properties

TEM Analysis



Pine wood biochar - 350°C

mag. 310 kX



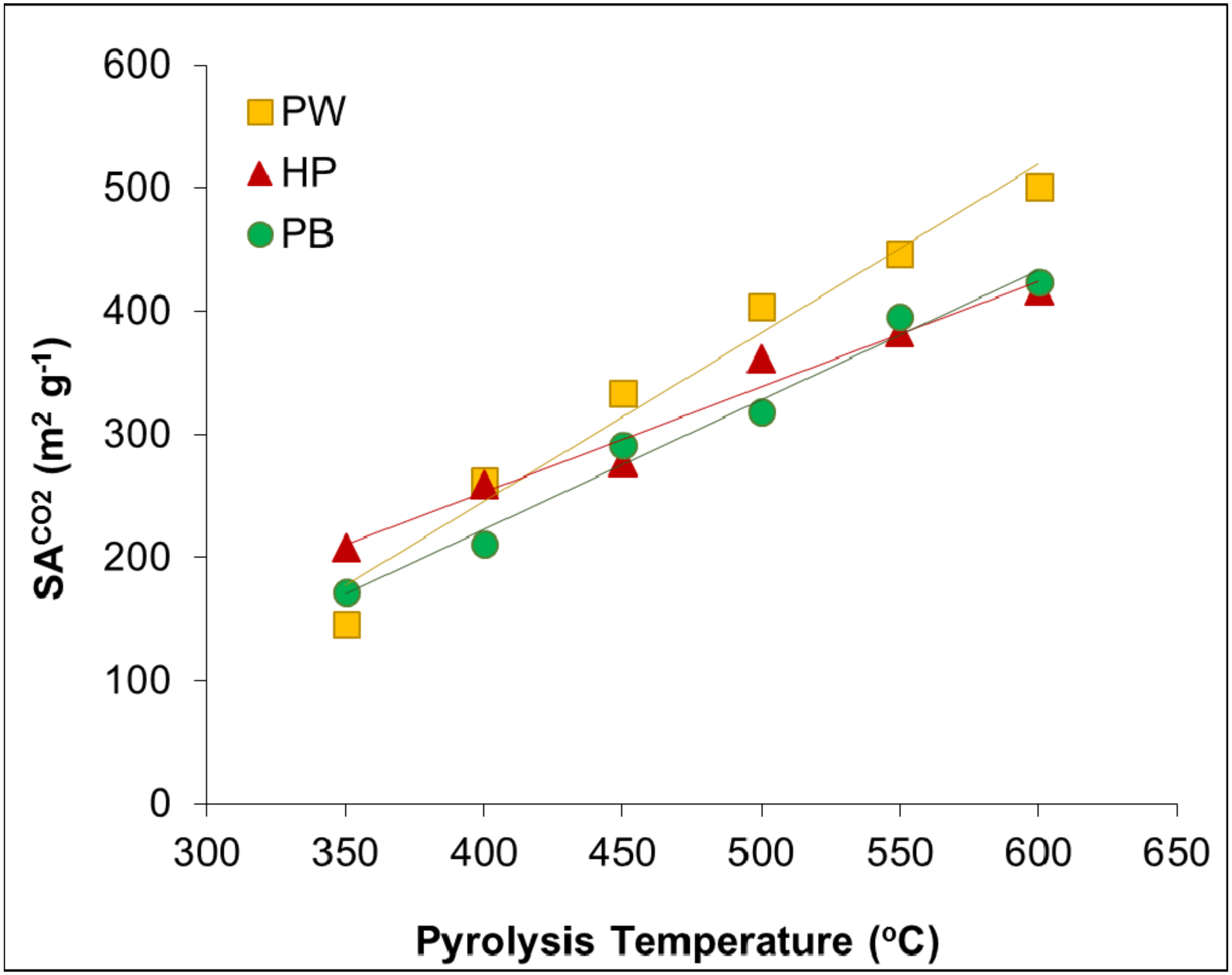
Pine wood biochar - 600°C

mag. 310 kX

Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

Biochar Properties

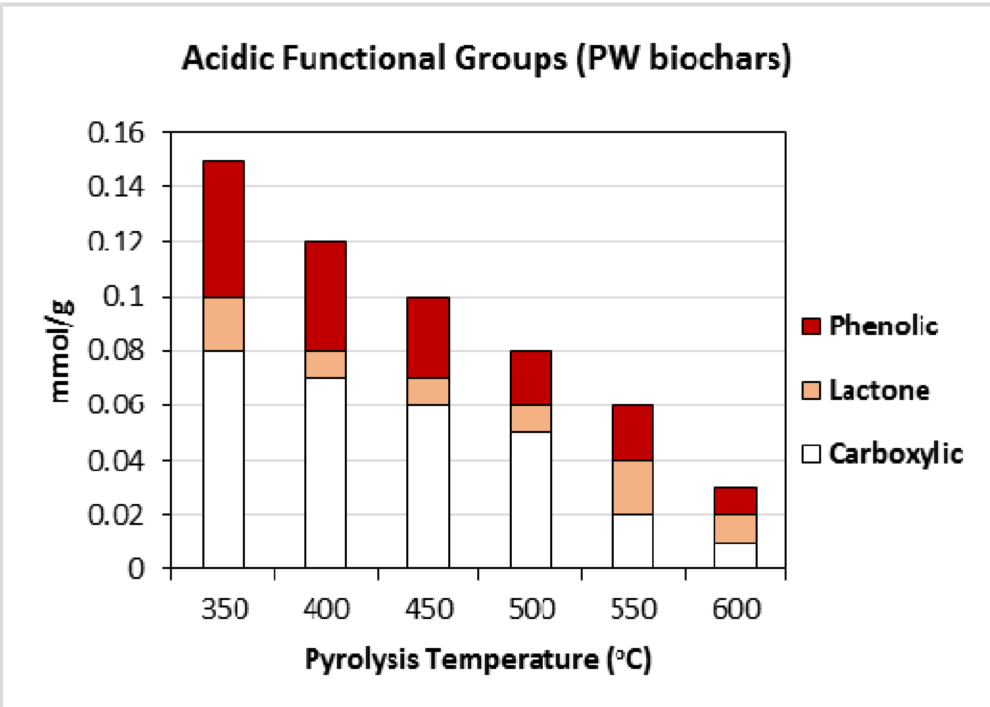
Surface Area

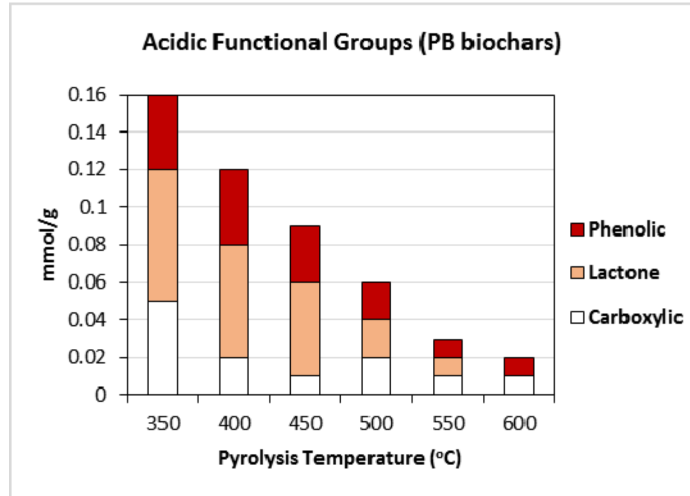


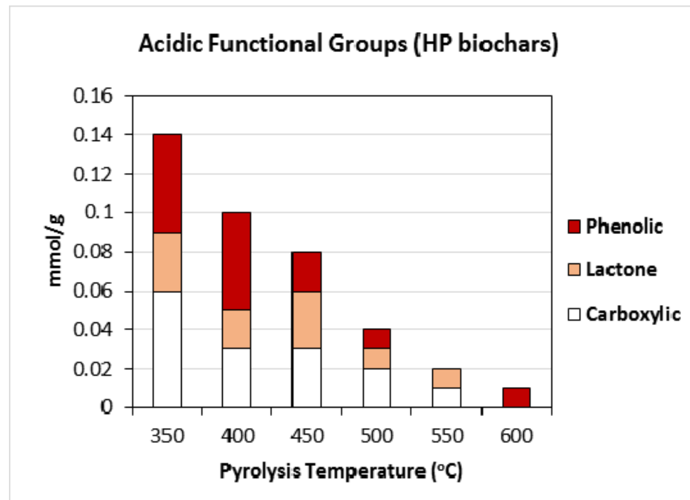
Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

Biochar Properties

Total Acidic Functional Groups (Boehm titration)

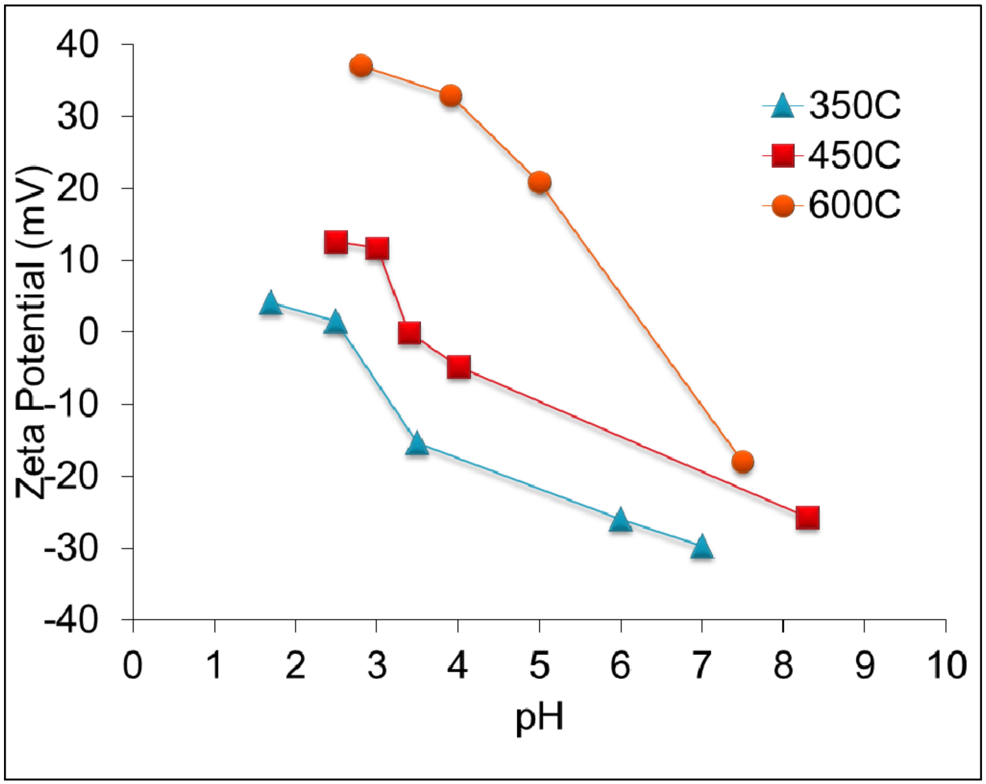


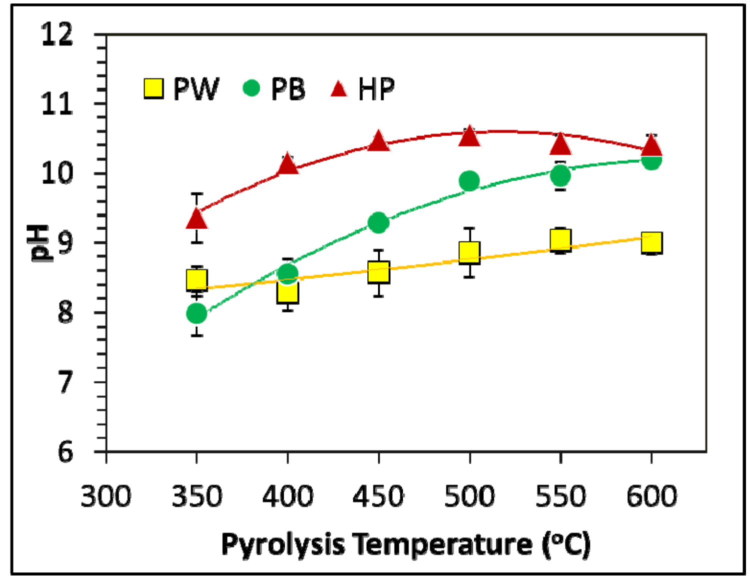


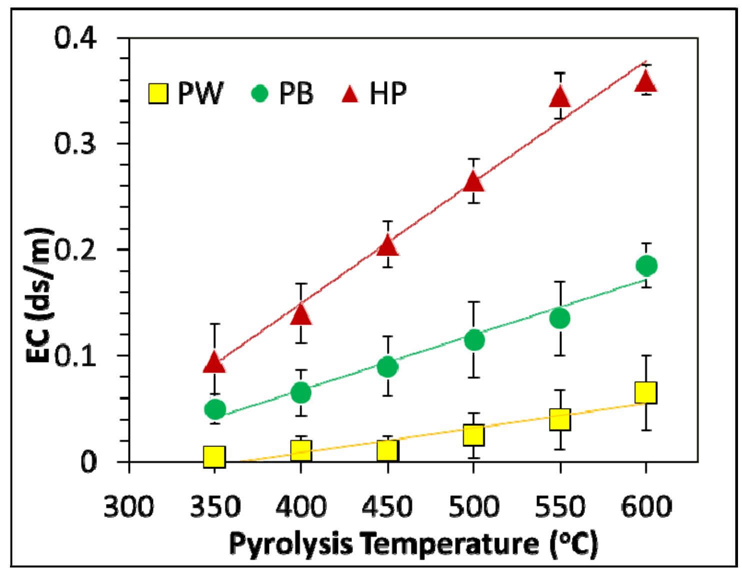


Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

Biochar Properties

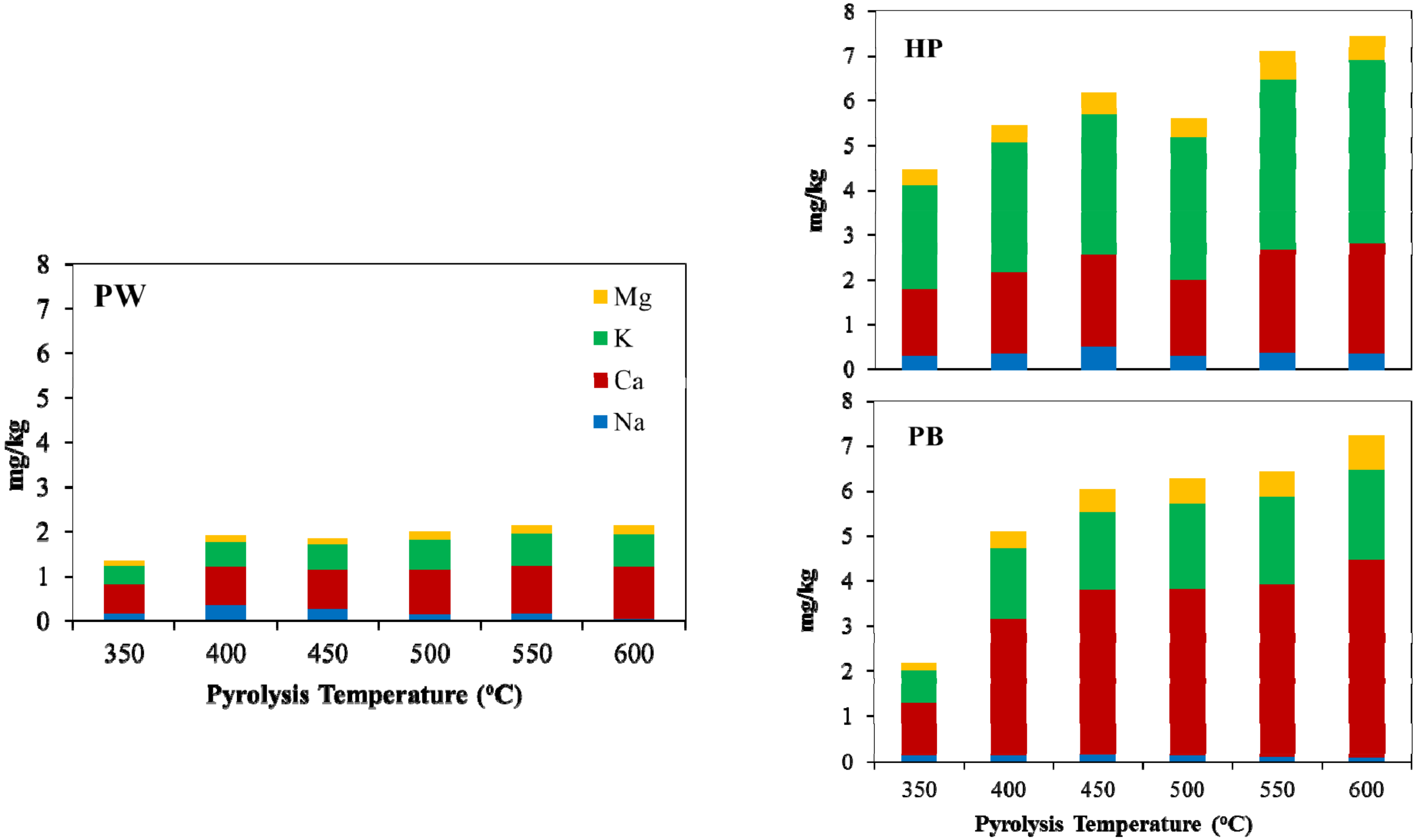






Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

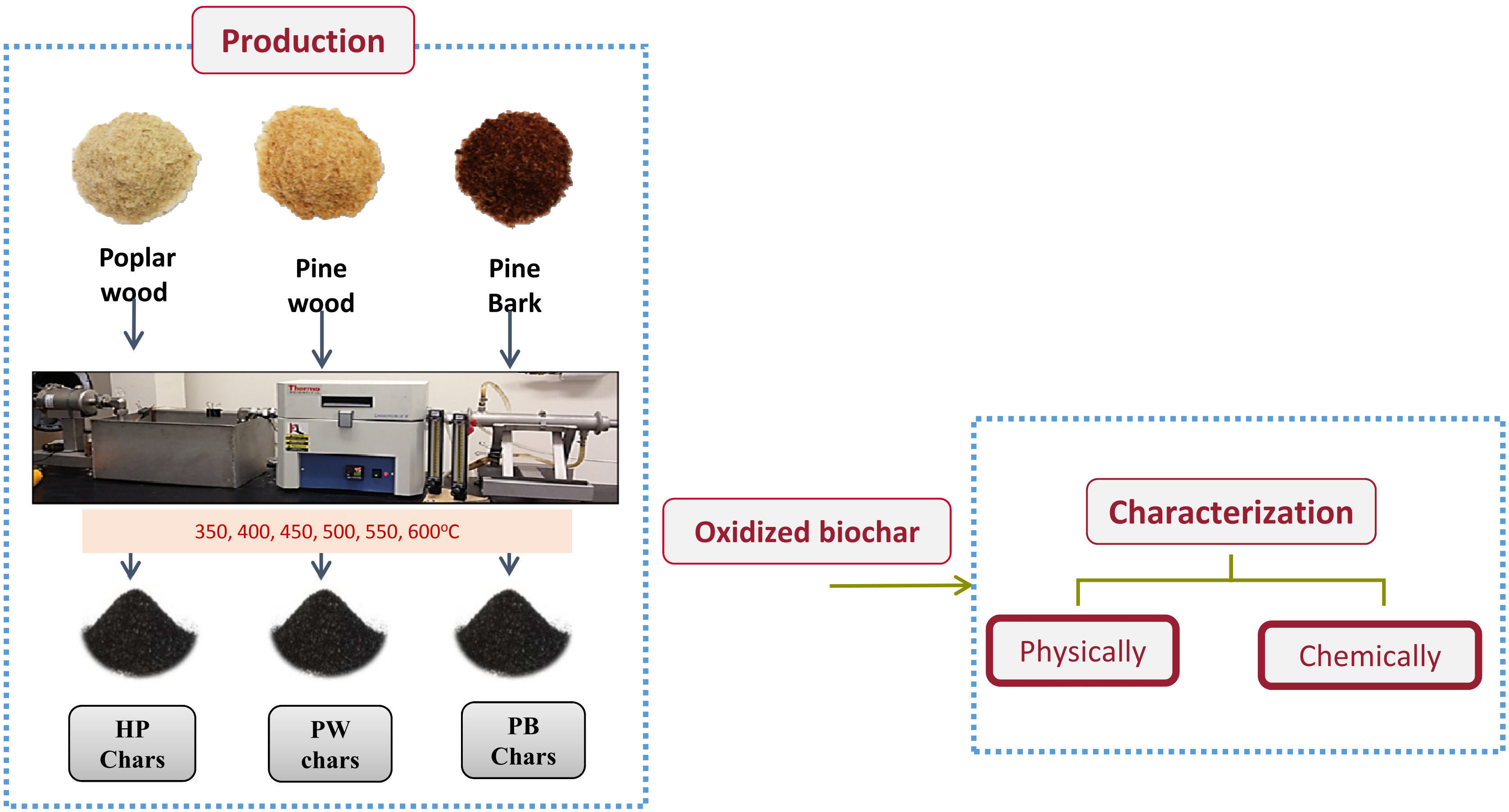
Biochar Properties



Suliman WSO, Harsh J,Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Influence of Feedstock Source and Pyrolysis Temperature on Biochar Bulk and Surface Properties. *Biomass and Bioenergy*, Volume 84, **2016,** pp 37-48

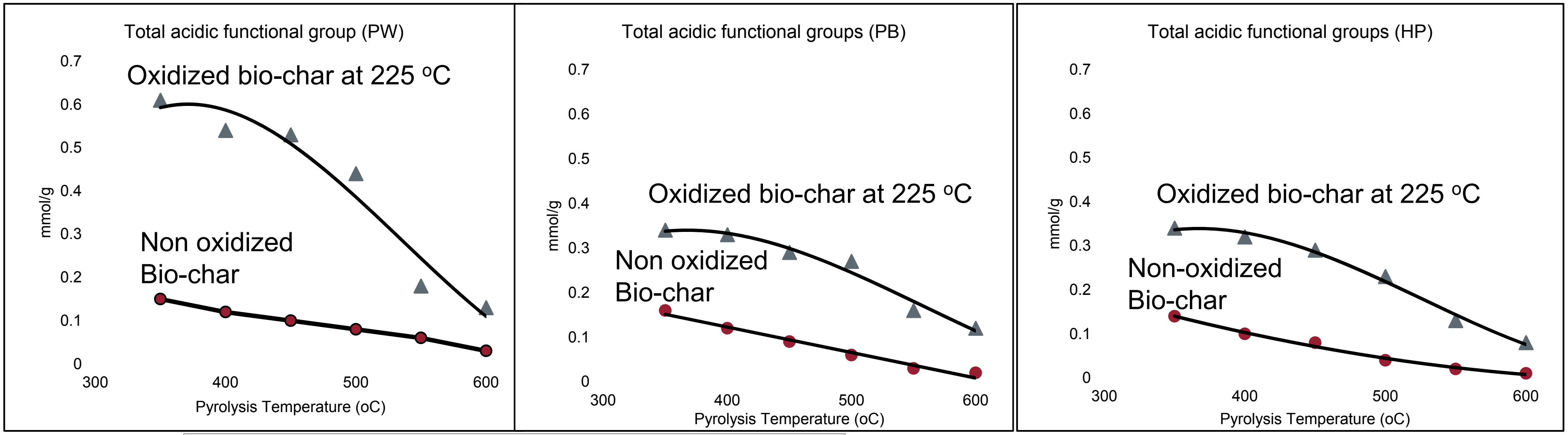
Biochar Properties

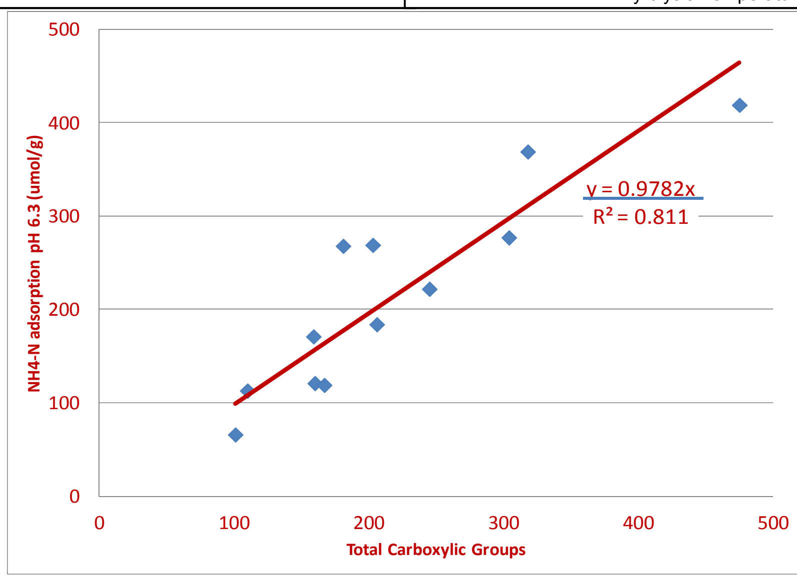
Study: Effect of Air Oxidation



Suliman W, Harsh JB, Abu-Lail NI, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Modification of Biochar Surface by Air Oxidation: Role of Pyrolysis Temperature. *Biomass and Bioenergy*, Vol.85, February **2016**, pp 1-11.

Biochar Properties





Cation exchange works well in systems without competitive cations.

Smith M, Ha S, Amonette JE, Dallmeyer I, Garcia-Perez M: Enhancing cation exchange capacity of chars through ozonation. Biomass and Bioenergy, 81, 2015, 304-314
Suliman W, Harsh JB, Abu-Lail N, Fortuna A-M, Dallmeyer I, Garcia-Perez M: Modification of biochar surface by air oxidation: Role of pyrolysis temperature. Biomass and Bioenergy, 85, 2016, 1-11

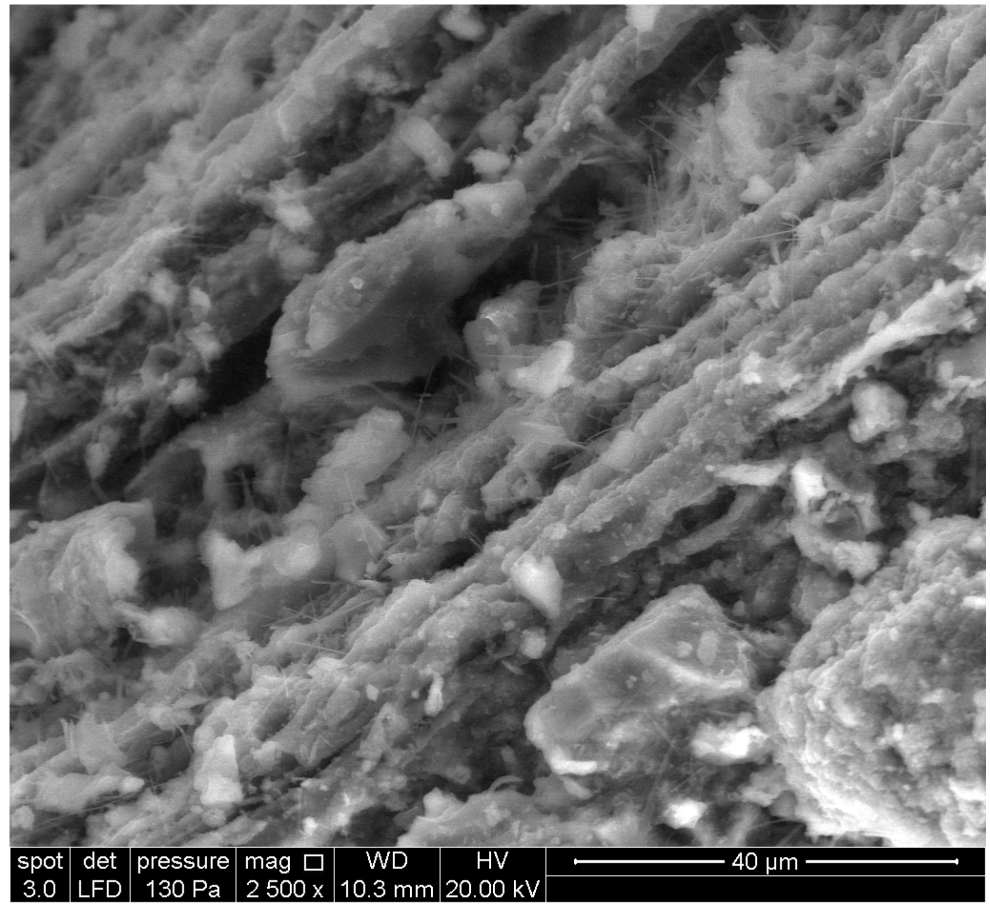
Environmental Services

Phosphate Precipitation

Precipitation of calcium for phosphate retention

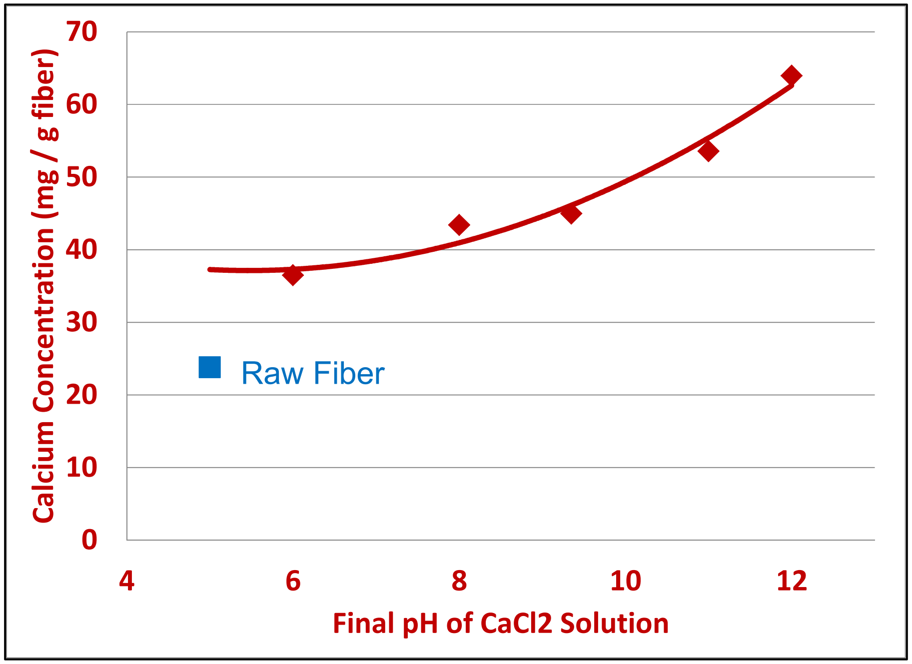
AD fiber was acid washed in 2% nitric acid and impregnated with calcium by immersion in a $CaCl_2$ solution followed by pH adjustment to 6, 8, 9.35, 11 and 12

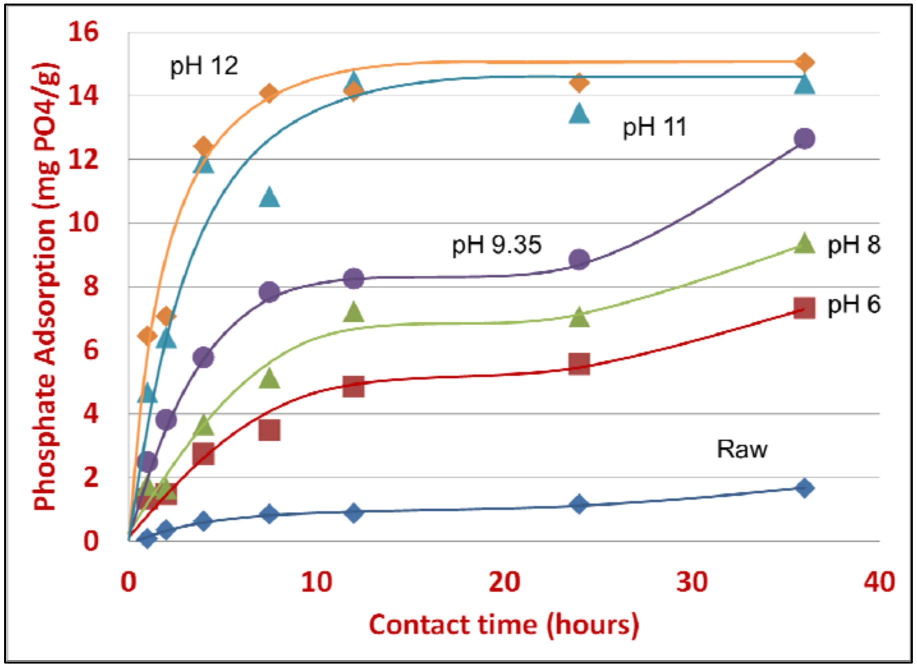
Modified fiber samples were then dried and pyrolized at 500^{o}C for 30 minutes using a spoon reactor



Environmental Services

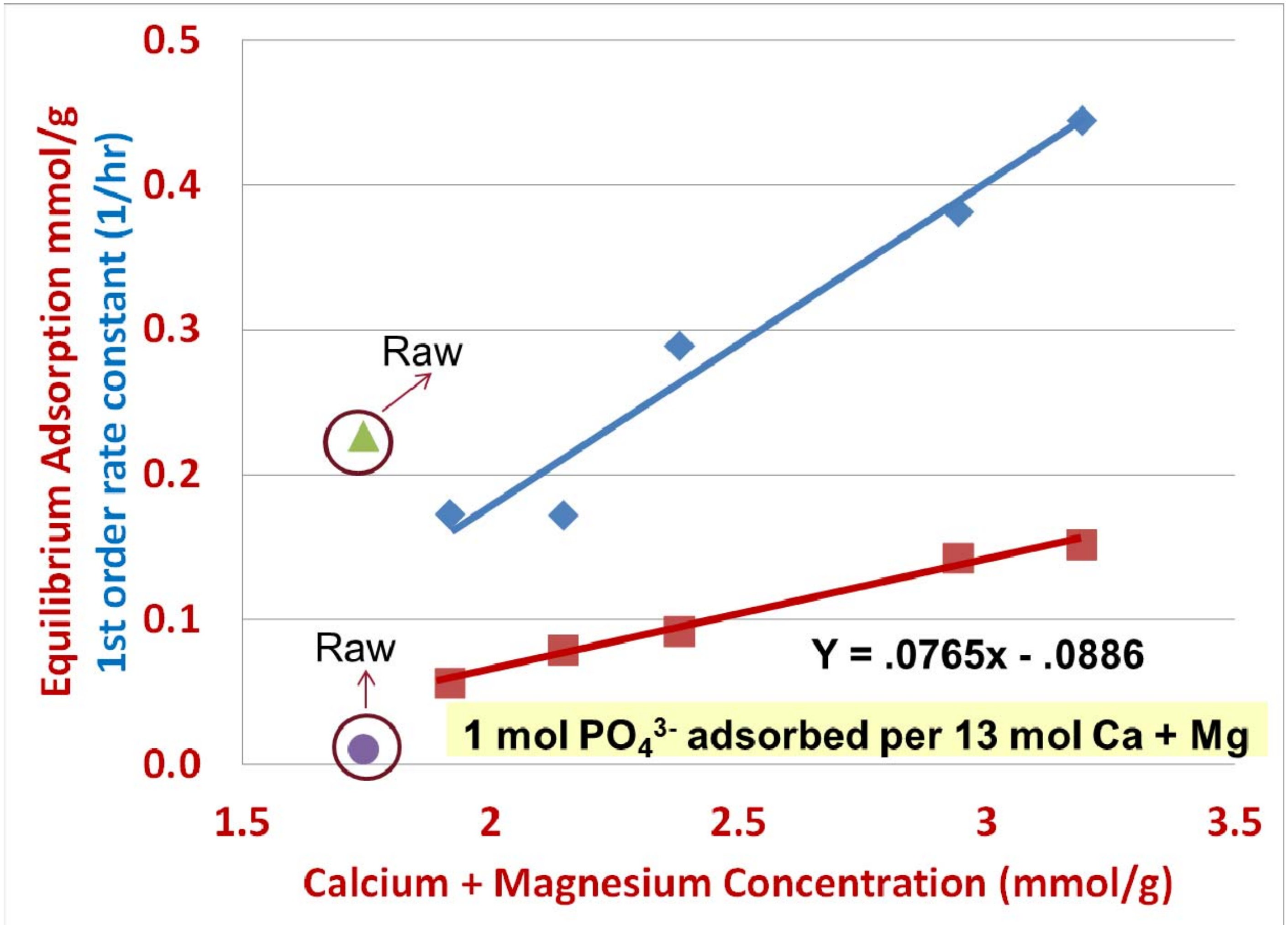
Pre-pyrolysis $CaCl_2$ modification of AD fiber





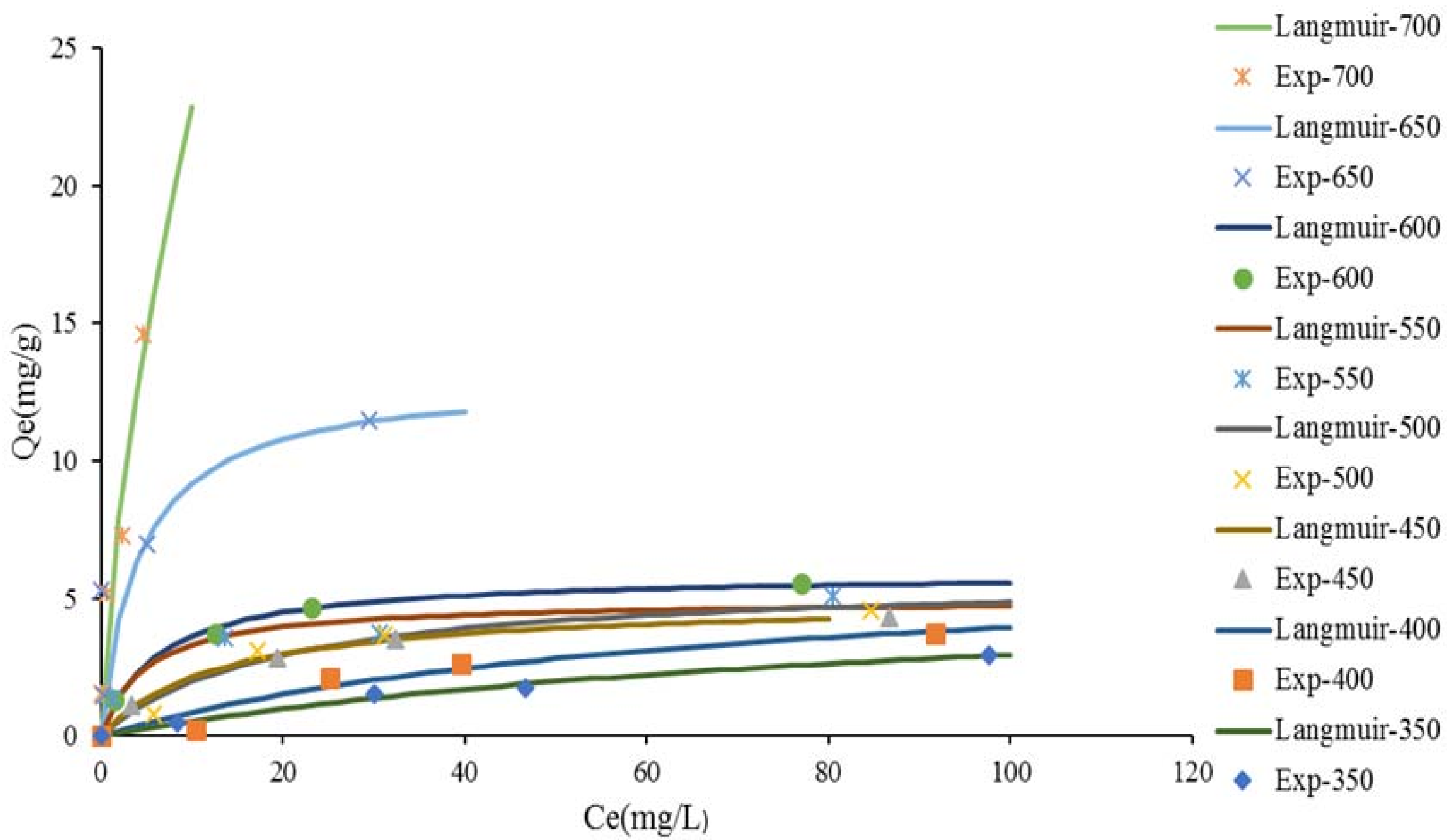
Environmental Services

Phosphorous Removal



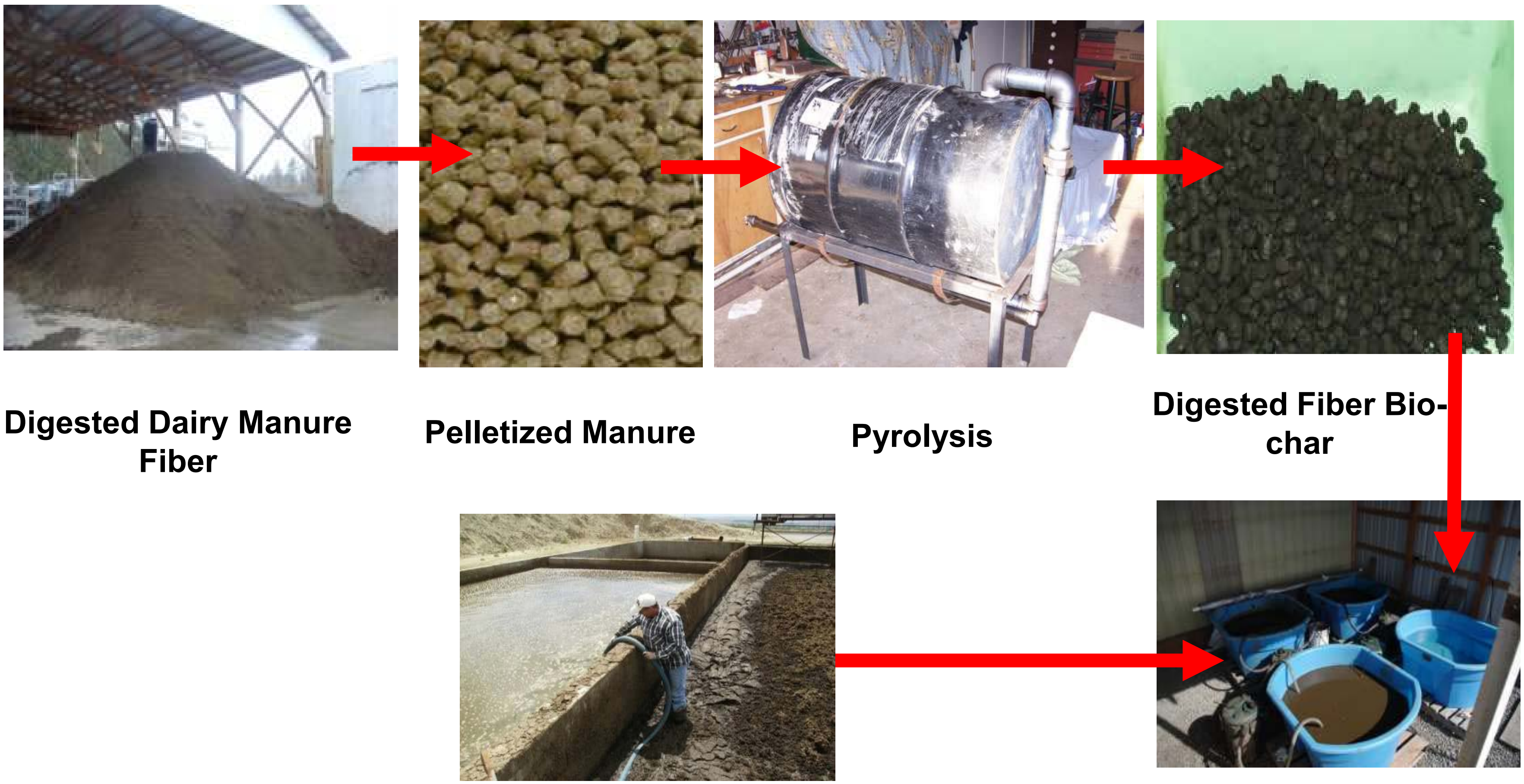
Environmental Services

Study: Adsorption isotherms of PO_4^{3-} on activated carbon produced from Anaerobic Digested Fiber at different temperatures



Environmental Services

Bio-chars for Phosphate Removal

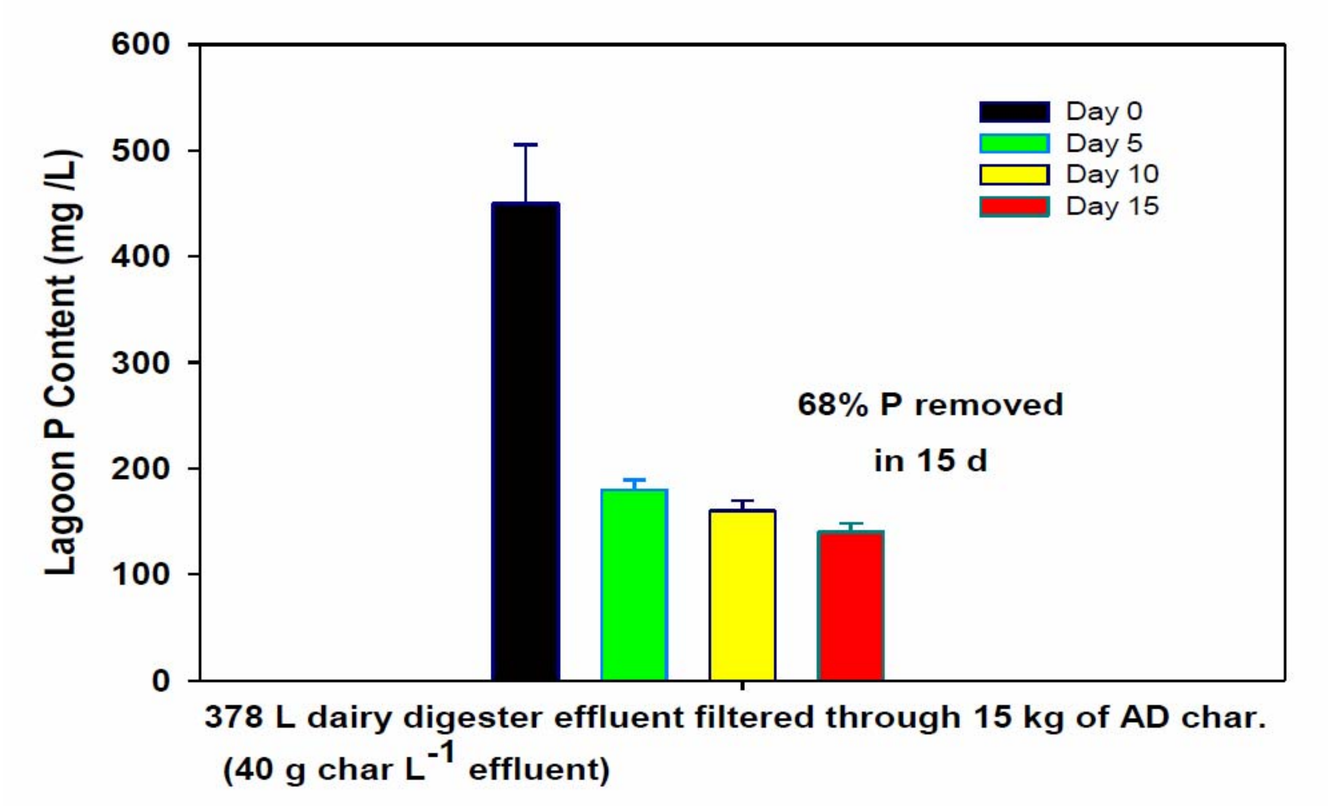


Streubel J, Kruger CE, Granatstein D, Collins H.P.: Bio-char Sorption of Phosphorus from Dairy Manure Lagoons. Future of Energy Conference. Seattle, WA, 2010

Streubel J, Biochar: Its characterization and utility for recovering phosphourous from Anaerobic Digested Dairy Effluent. PhD dissertation WSU, May 2011.

Environmental Services

Bio-chars for Phosphate Removal

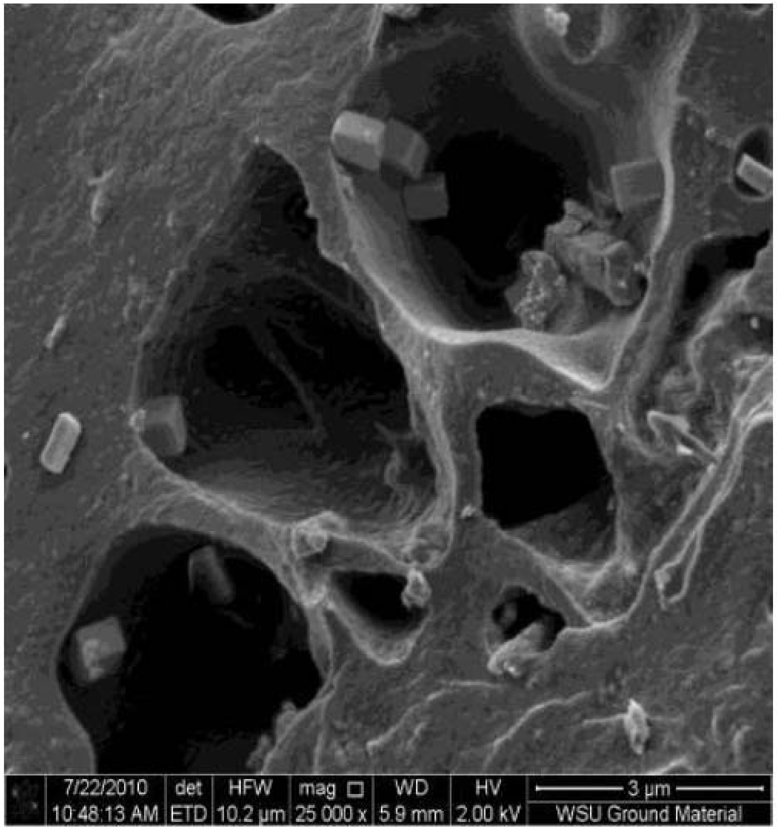


Anaerobic digested bio-char can be effectively used to reduce phosphorous from dairy lagoons.

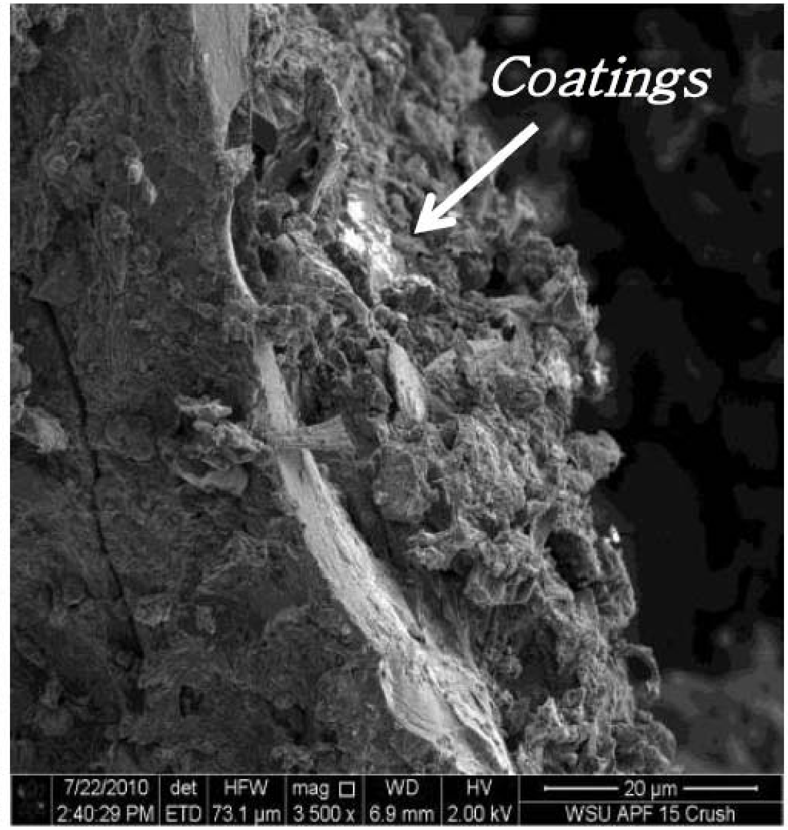
Streubel J, Kruger CE, Granatstein D, Collins H.P.: Bio-char Sorption of Phosphorus from Dairy Manure Lagoons. Future of Energy Conference. Seattle, WA, 2010

Streubel J, Biochar: Its characterization and utility for recovering phosphourous from Anaerobic Digested Dairy Effluent. PhD dissertation WSU, May 2011.

Environmental Services



Un-Amended Char



Lagoon-treated

Most of the phosphorous removed seems to be in the colloidal form

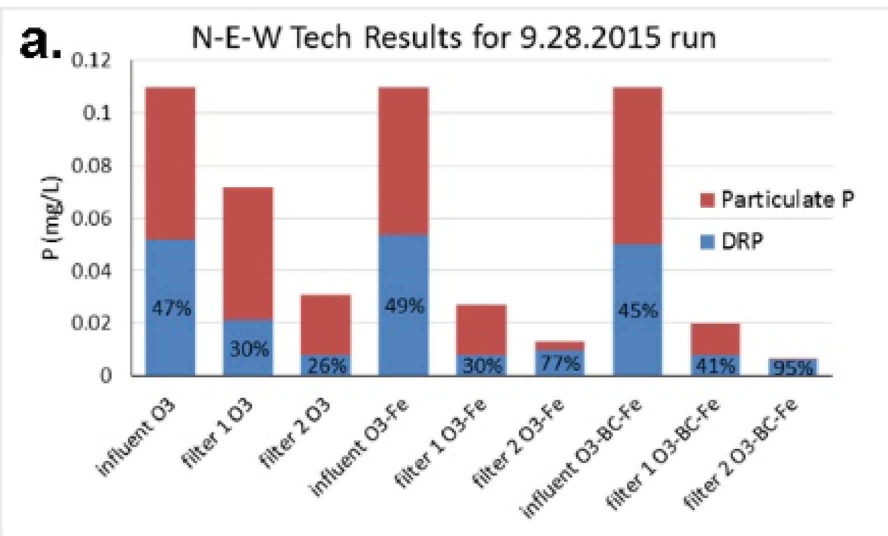
Streubel J, Kruger CE, Granatstein D, Collins H.P.: Bio-char Sorption of Phosphorus from Dairy Manure Lagoons. Future of Energy Conference. Seattle, WA, 2010

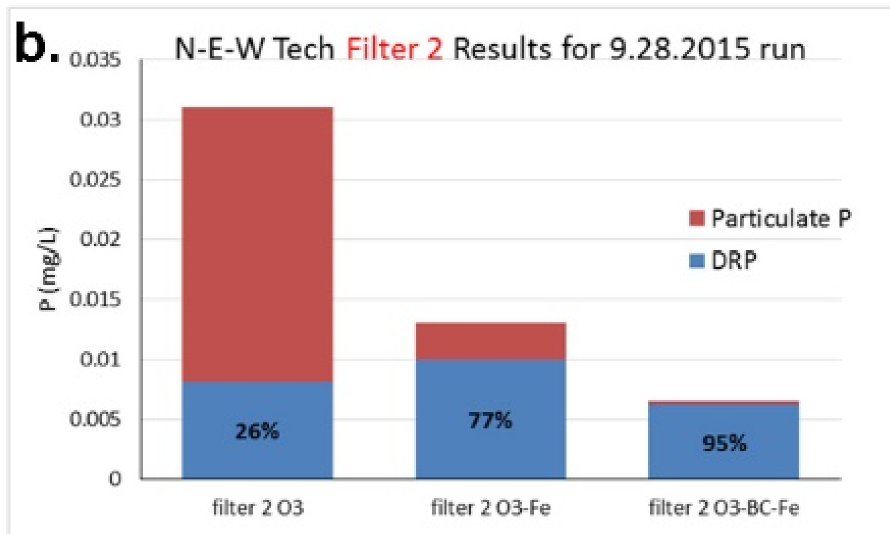
Streubel J, Biochar: Its characterization and utility for recovering phosphourous from Anaerobic Digested Dairy Effluent. PhD dissertation WSU, May 2011.

Engineered Biochars

Phosphorous Removal

Prof. Greg Moller UI



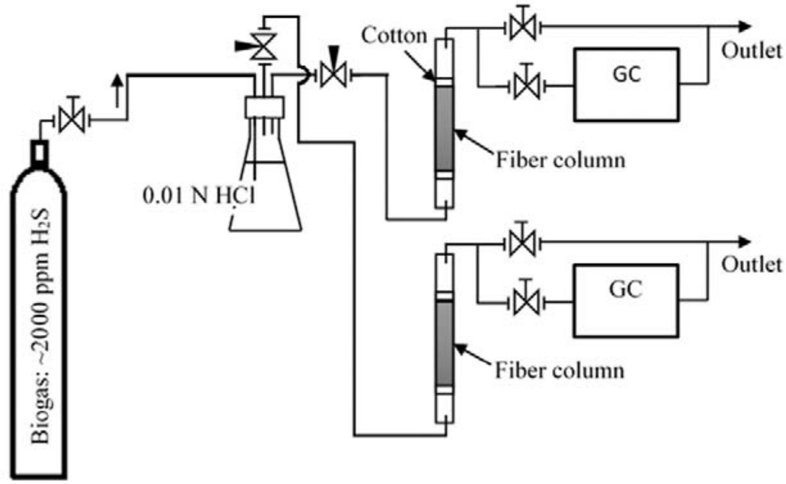


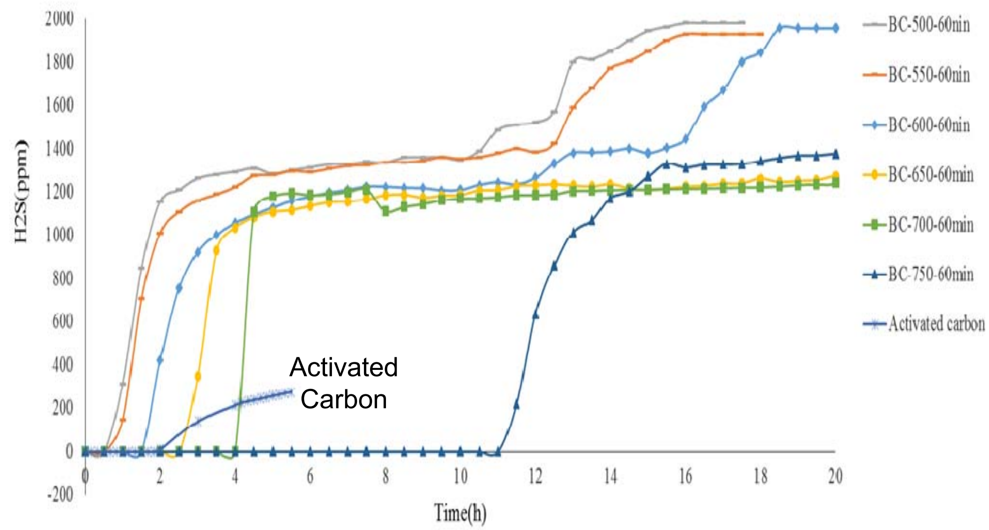
DRP: Dissolved Reactive Phosphorous

Greg Moller: N-E-W TechTM: Design-build of an intensification resource recovery (IR2) technology at the Nutrient, Energy, Water nexus. University of Idaho

Environmental Services

Study: H_2S removal from biogas with Anaerobic digested fiber





Bio-char derived from anaerobic digested fiber can be an excellent adsorbent for H_2S removal from biogas.

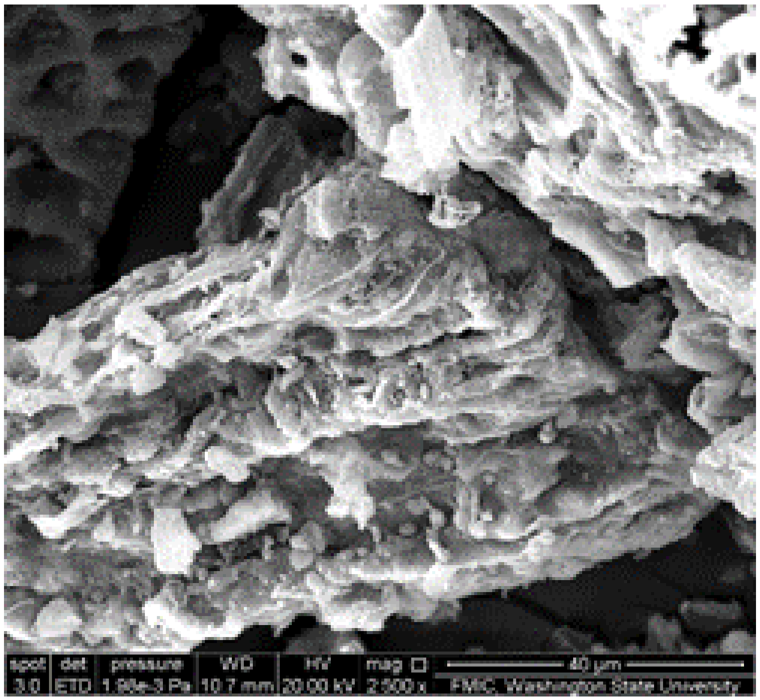
The resulting biochar contains 10-28 % of Sulfur on the surface

Pelaez-Samaniego MR, Smith MW, Zhao QZ, Garcia-Perez T, Frear C, Garcia-Perez M: Charcoal from Anaerobically digested dairy fiber for removal of hydrogen sulfide within biogas. In preparation, 2017

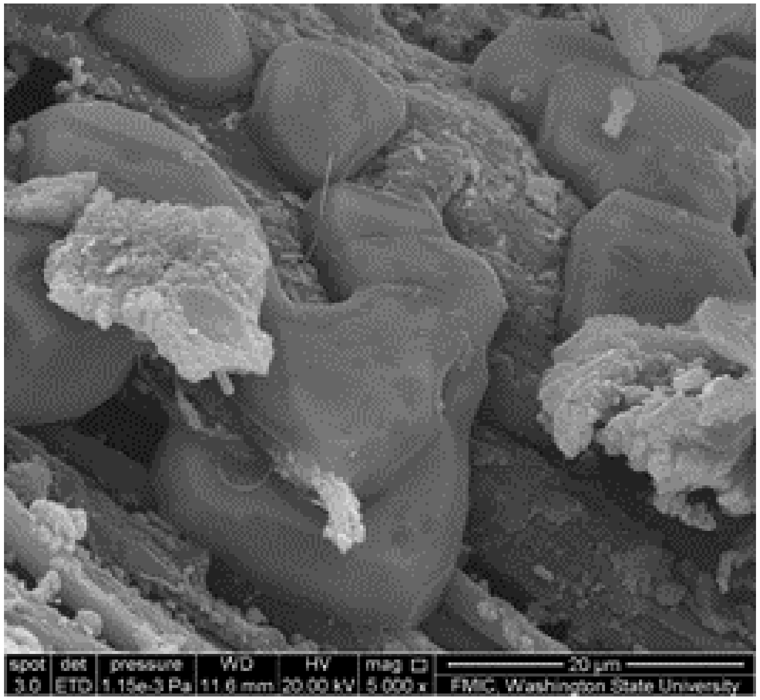
Environmental Services

Study: H_2S removal from biogas with Anaerobic digested fiber

Char



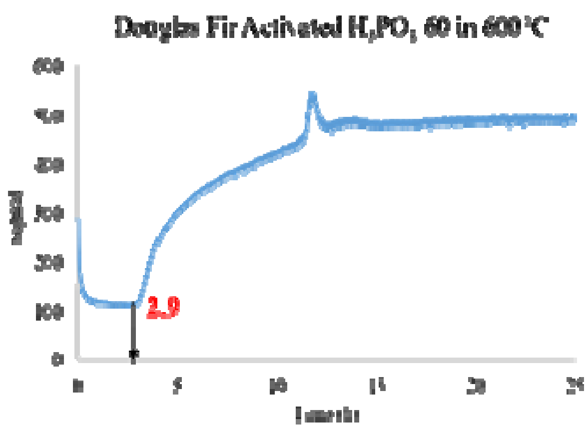
S on char surface after H_2S adsorption

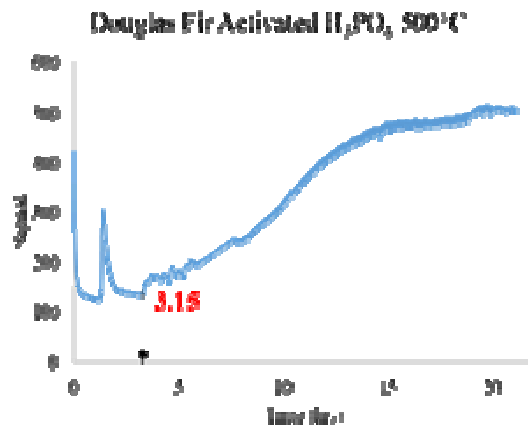


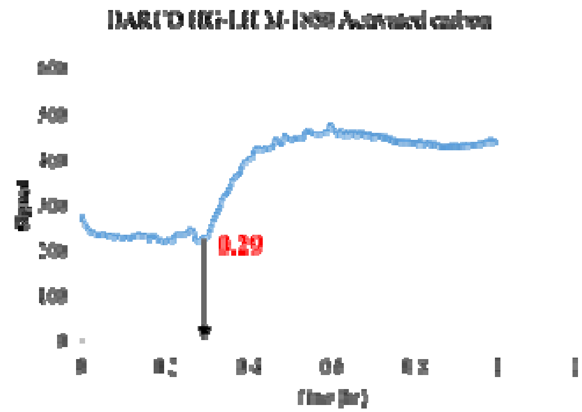
Pelaez-Samaniego MR, Smith MW, Zhao QZ, Garcia-Perez T, Frear C, Garcia-Perez M: Charcoal from Anaerobically digested dairy fiber for removal of hydrogen sulfide within biogas. In preparation, 2017

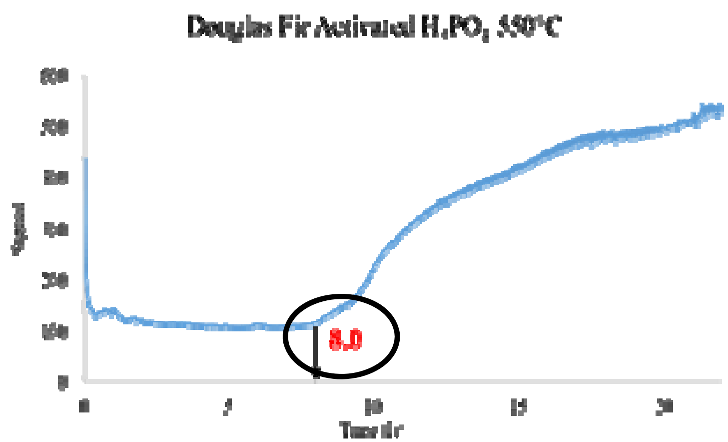
Environmental Services

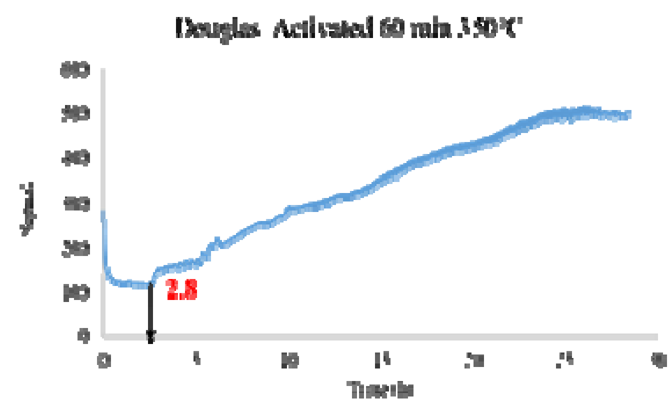
Study: Ammonia adsorption breakthrough curves of biochar produced at different temperature (DF Chemically Activated Carbon (with H_3PO_4))







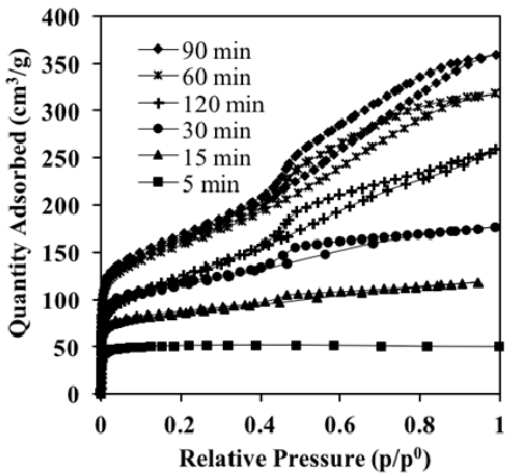




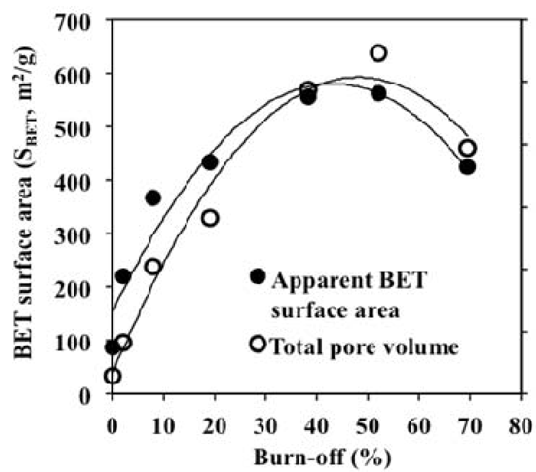
Environmental Services

Physical activation of a novel type of lignin (sulfite-pretreated, saccharified Douglas Fir forest harvest residues (FRS) (CO_2 activation at 700 °C).

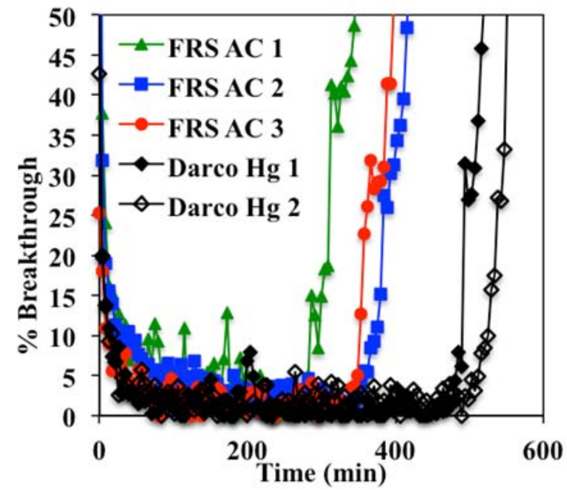
N_2 adsorption-desorption isotherms



Optimal Activation Conditions



Breakthrough curves of mercury



Summary of adsorption results

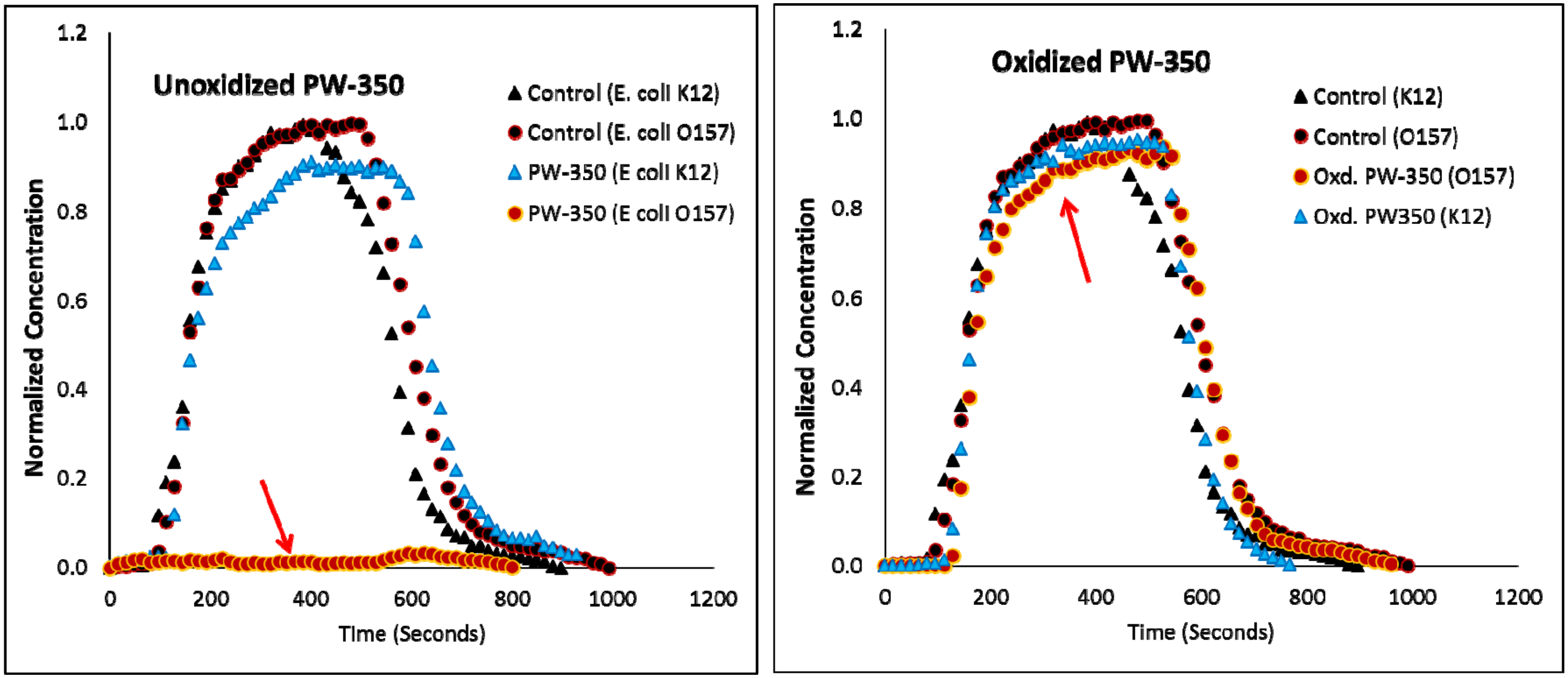
Carbon	S_{BET} (m^2/g)	V_{total} (cm^3/g)	V_{meso} (cm^3/g)	V_{micro} (cm^3/g)	Inlet [Hg^0] in flue gas (µg Hg^0/Nm^3)	Equilibrium Hg^0 Adsorption Capacity (µg Hg^0/g AC) @ 50 µg Hg/Nm^3	Average Percent Hg^0 Removed (%)
Darco Hg	660	0.718	0.474	0.209	21.8	1133	98.5
Darco Hg	660	0.718	0.474	0.209	26.5	1226	97.8
FRS AC 1	659	0.621	0.402	0.188	27.0	674	95.1
FRS AC 2	686	0.670	0.442	0.191	25.9	863	96.0
FRS AC 3	682	0.665	0.440	0.189	21.8	857	97.6
Average (FRS AC)	**676**	**0.652**	**0.428**	**0.189**	**24.9**	**798**	**96.2**

FRS AC: Chars activated at 700 °C, under CO_2 for 90 min.

Dallmeyer I, Fish D, Fox C, Spink T, Smith M, Garcia-Perez M, Wolcott M: Mesoporous Activated Carbon from Softwood SPORL Lignin and Its Implication in Vapor-Phase Mercury Capture. Paper in preparation, 2017

Environmental Services

Effects of air oxidation on e-coli removal

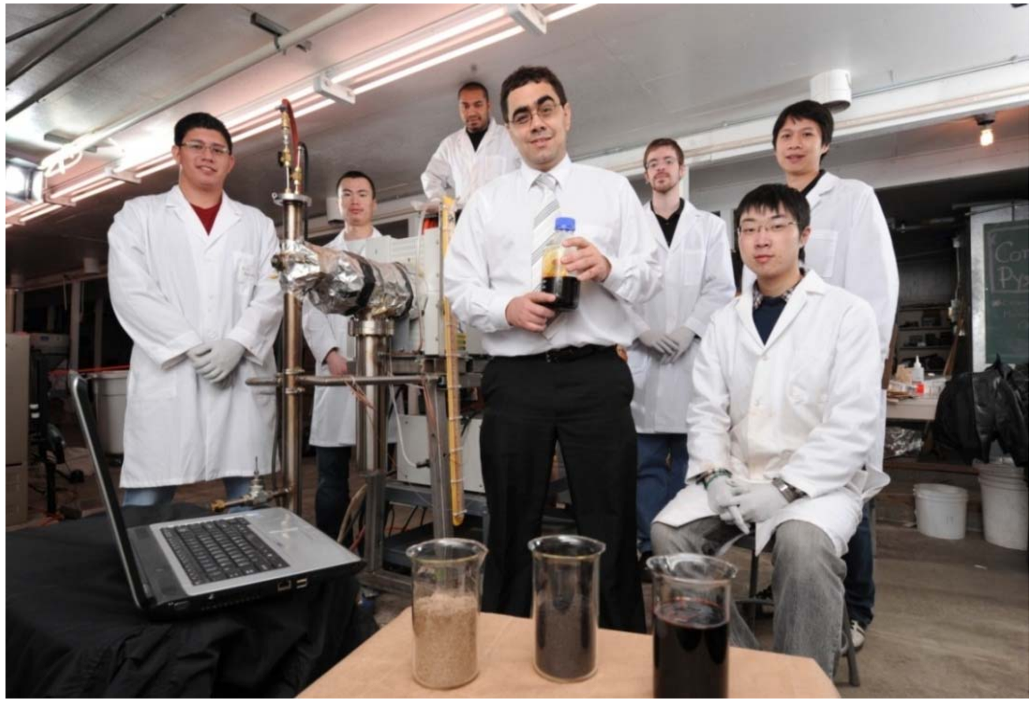


Suliman W, Harsh J, Fortuna A, Garcia-Perez M, Abu-Lail N: Towards the quantification of the effects of biochar oxidation and pyrolysis temperature on the transport of pathogenic and nonpathogenic E. coli in biochar amended sand columns (Submitted to *Environmental Science and Technology Journal*, **2016**)

Conclusions

- We have proposed a new model of AD biorefinery that make use of engineered biochars for nutrients removal.
- The role of liquid intermediate as a very reactive phase responsible for many of the important reactions encountered during biomass carbonization was discussed.
- Bio-char properties depend on the feedstock used, pyrolysis conditions and post-pyrolysis treatment.
- Bio-chars capable of adsorbing phosphate, ammonia, H_2S, e-coli and mercury were developed from relevant lignocellulosic waste streams.

ACKNOWLEDGEMENT



We would like to thank the funding agencies supporting my Research Program

WASHINGTON STATE UNIVERSITY AGRICULTURAL RESEARCH CENTER

U.S. NATIONAL SCIENCE FOUNDATION

WASHINGTON STATE DEPARTMENT OF AGRICULTURE

SUN GRANT INITIATIVE, U.S. DEPARTMENT OF TRANSPORTATION, USDA

WASHINGTON STATE DEPARTMENT OF ECOLOGY

U.S. DEPARTMENT OF ENERGY

Thank you ☺

Questions?