



UPCYCLING OF BIOWASTES FOR ENERGY AND MATERIALS: A CIRCULAR ECONOMY APPROACH

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University Research Leadership Chair

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BRIL-OMAFRA KTT Workshop March 22, 2024, Guelph, ON



Bio-Renewable Innovation Lab (BRIL)

BRIL is a multidisciplinary research facility initiated in 2014

Mission: To conduct research and develop innovative technology solutions to help circular economy for long-term sustainability.

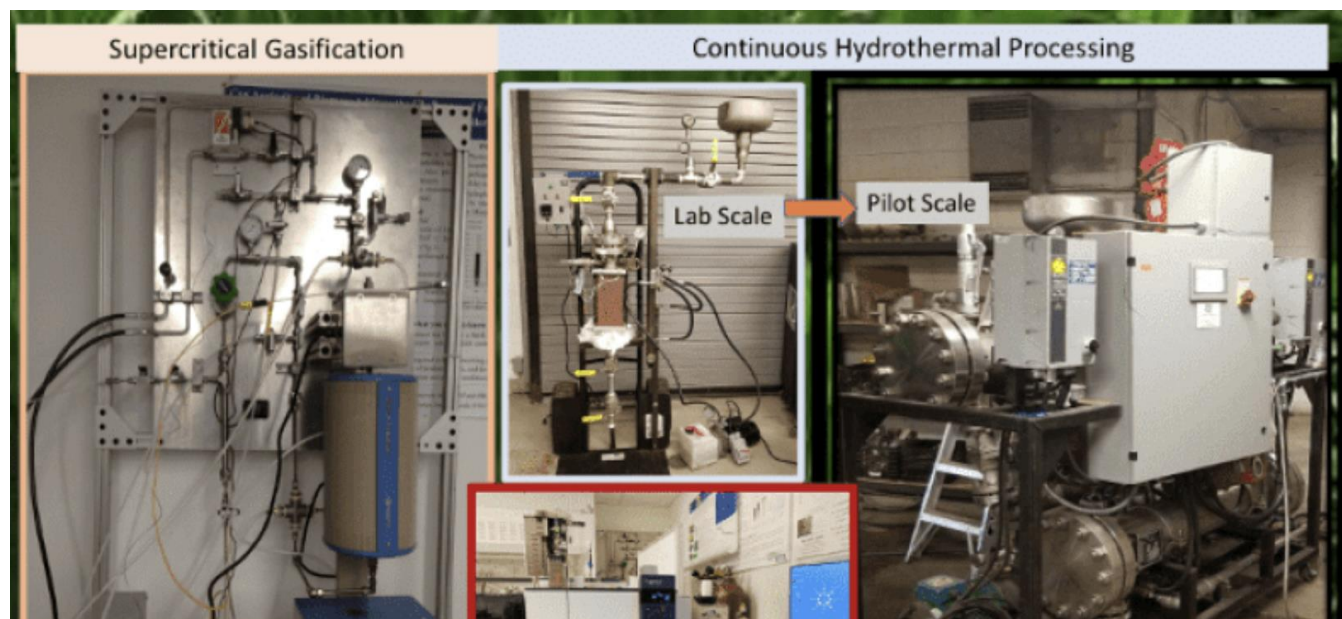
Research Focus: Valorisation of various organic wastes to promote sustainability through circular economy concept

Other Activities:

- Knowledge Translation and Transfer (KTT)
 - Webinars
 - Workshop

- HQP Training
 - Undergrad, Graduate,
 - Post doctoral

- Research Capacity Building



Process Technologies	Analytical Capabilities
Thermochemical Hydrothermal Supercritical Modeling & Scaling-up	Py- GC/MS HP – TGA, TGA – FTIR Surface and pore Characterization Catalysis and Kinetics Studies

- Bio-Renewable Innovation Lab (BRIL) at Guelph is built and located in the Thornbrough Building of University of Guelph.
- BRIL composed of two parts: research pilot plant and analytical laboratory.

Research Pilot Plant

The pilot plant involves research facilities such as supercritical, chemical looping, multi-stage, and circulating fluidized bed reactors, which have helped our group achieving research in three different areas of biomass conversion.

1) Feedstocks pre-processing



Ultrasonic equipment



Pelletization

2) Thermochemical conversion processes



Hydrothermal processing



Moving bed reactor



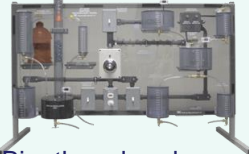
Chemical Looping Gasifier



3) Bio-chemical conversion processes



Bio-syngas Fermentation



Bioethanol and biodiesel facility



Supercritical flow reactor



Multi-Stage Gasifier



Two-Stage Gasifier-combustor

Analytical Laboratory

Scientific analytical instruments for both qualification and quantification analyses.



Elemental Analyzer (C, H, N, S, O)



GC-FID/TCD



FTIR



DSC-TGA



Bomb Calorimeter



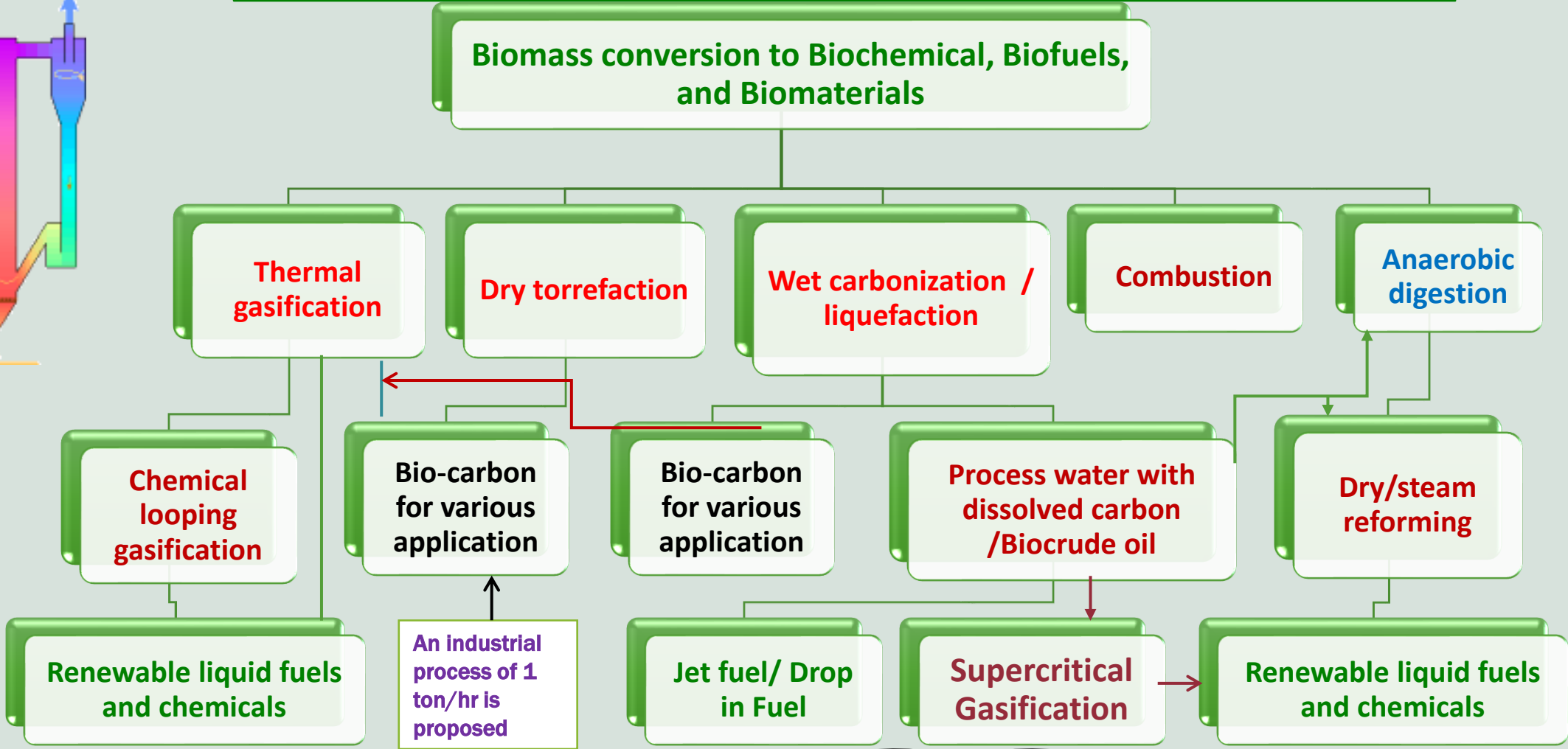
Planetary Ball Mill (Grinder)



PY-Pyrolizer with GCMS

BIOWASTE CONVERSION RESEARCH PROGRAM AT BRIL

“WASTE IS A RESOURCE-WAITING FOR AN OPPORTUNITY”



Life Cycle analysis (LCA) and Life Cycle cost analysis (LCAA)

Our Ultimate Goal is to Develop

“A wide variety of renewable products including bio-carbon, renewable chemicals, bio-methane, and bio-fertilizers from a variety of **non-food sustainable agri-food wastes** feedstocks.”

Meeting Goal by

- Valorization of agricultural and food wastes (crop residues, greenhouse, and food processing wastes) that has the potential to replace non-renewable fossil based resources
- Developing green processes and products value chain to mitigate GHG emissions
- Strengthening sustainability and bioeconomy of Canadian agriculture and agri-food sector

RESEARCH OVERVIEW

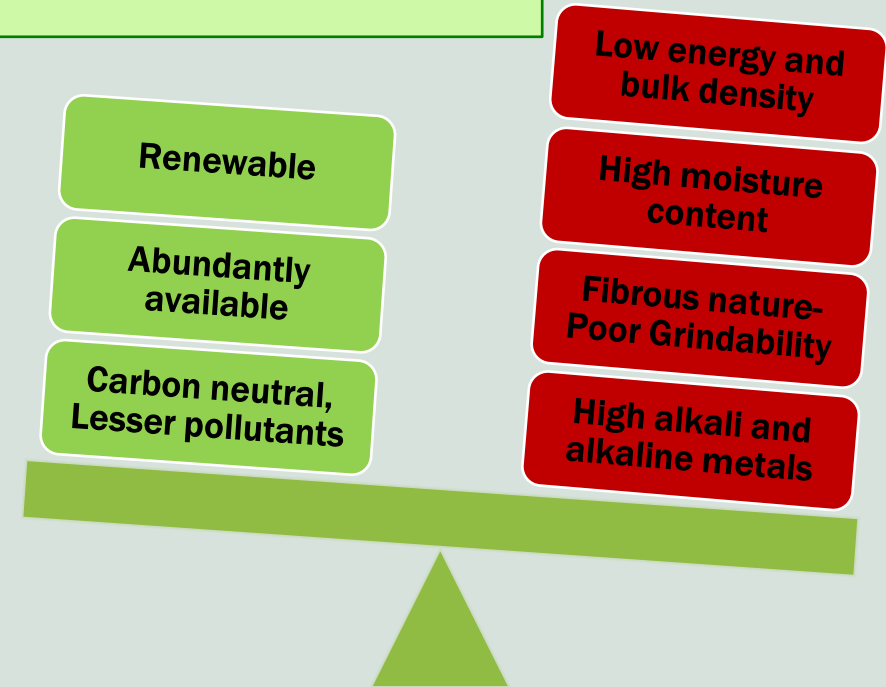
THEME: “WASTE IS A RESOURCE - WAITING FOR AN OPPORTUNITY”



“Agri-food wastes is not regarded as an ideal replacement for fuel and materials application”

Hydrothermal Carbonization (HTC) processing where biomass is treated with hot compressed water instead of drying exhibits unique physicochemical properties

HTC products from low quality agri-food residue can be a potential newer value chain

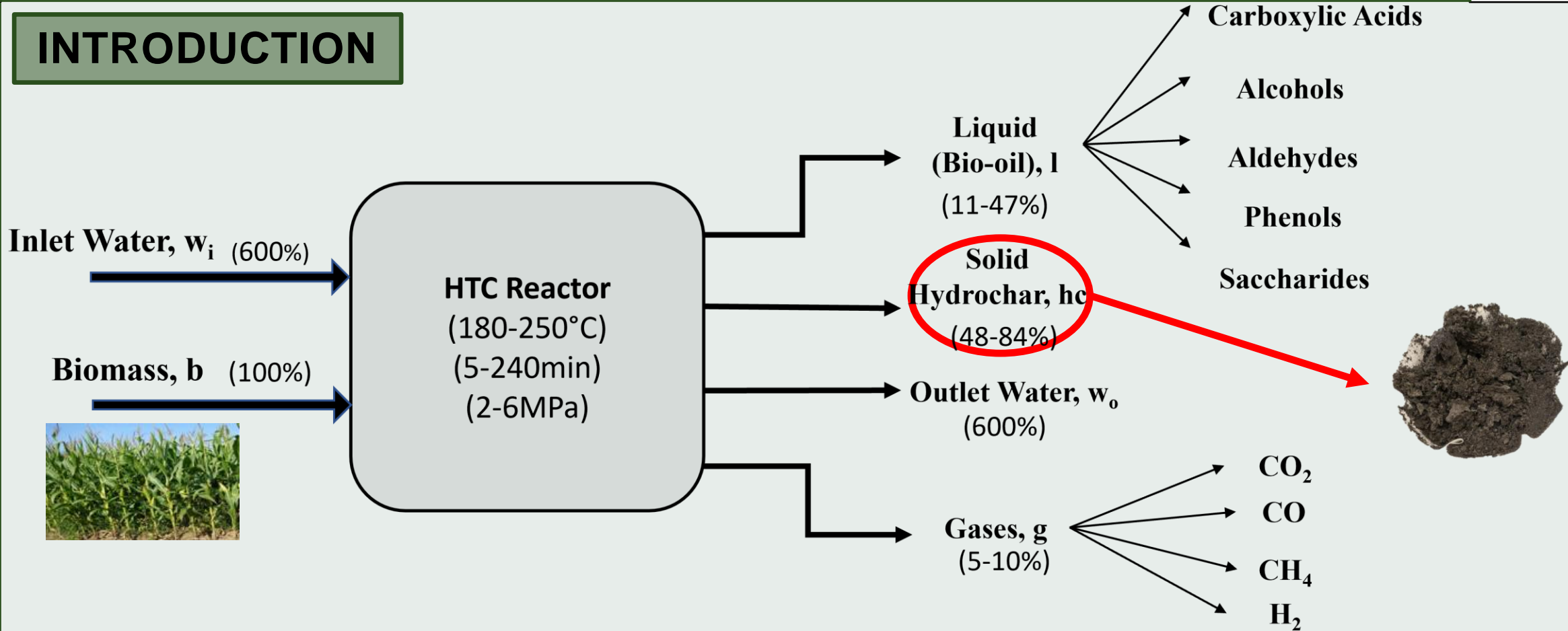


Research Questions:

- Can we produce industrial grade biocarbon from this low quality biomass (low alkali metals, higher HHV, and higher grindability) for energy and materials applications?
- Will there be any industrial grade biochemical as a co-product from HTC Process Water (HTCPW)?

Hydrothermal Carbonization

INTRODUCTION

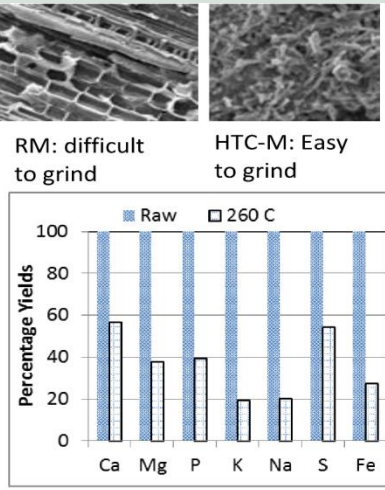
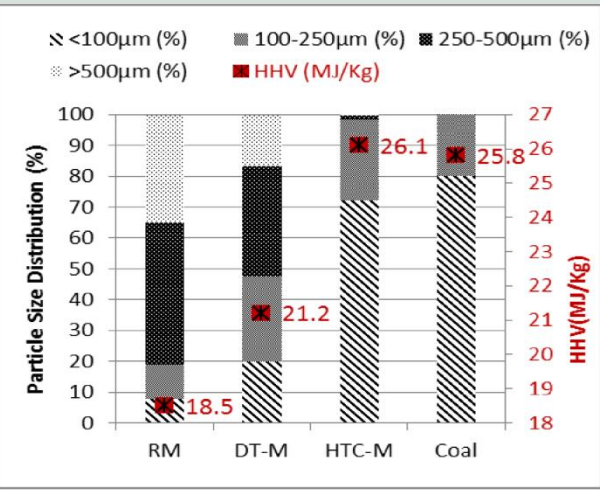
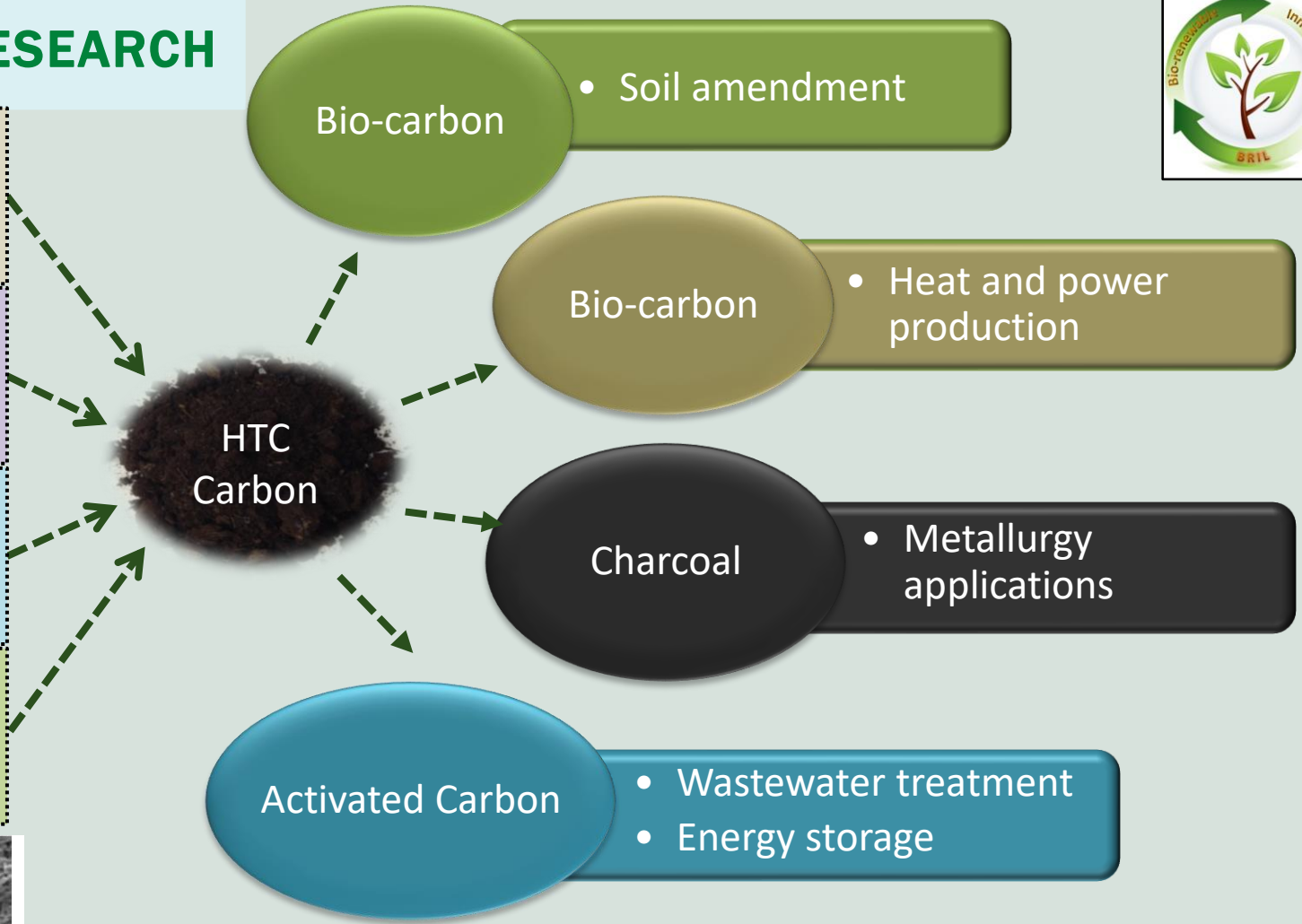


- ### Challenges Addressed
- Eliminates Drying Step
 - Accommodate Biomass Variability
 - Reduce Ash/Impurities
 - Simple Process

BIOCARBON DEVELOPMENT RESEARCH



Woody Biomass		Construction Waste	
	Willow		
Purpose Grown Biomass		Miscanthus	
	Switchgrass		
Agricultural Biomass		Wheat Straw	
	Corn Stover		
Wet Biomass		Tomato Vines	
	Corn Husks		

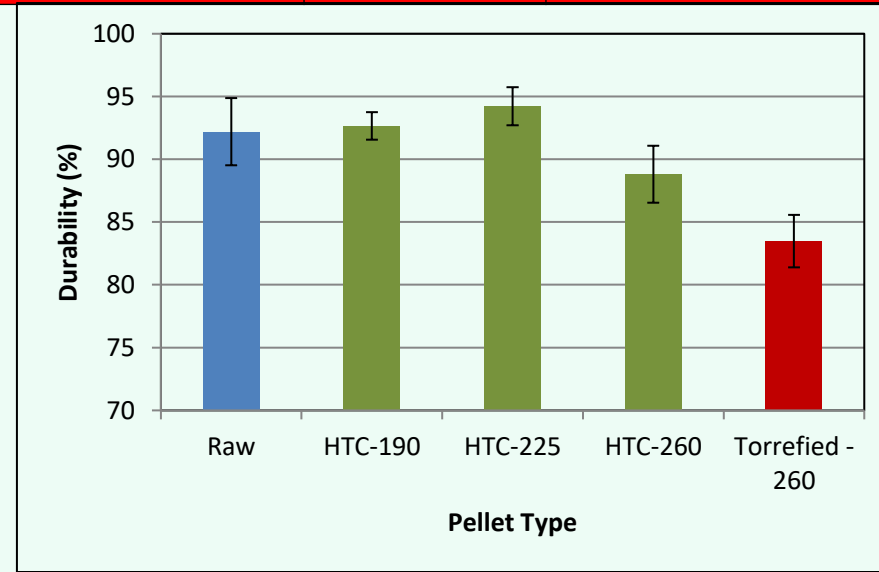
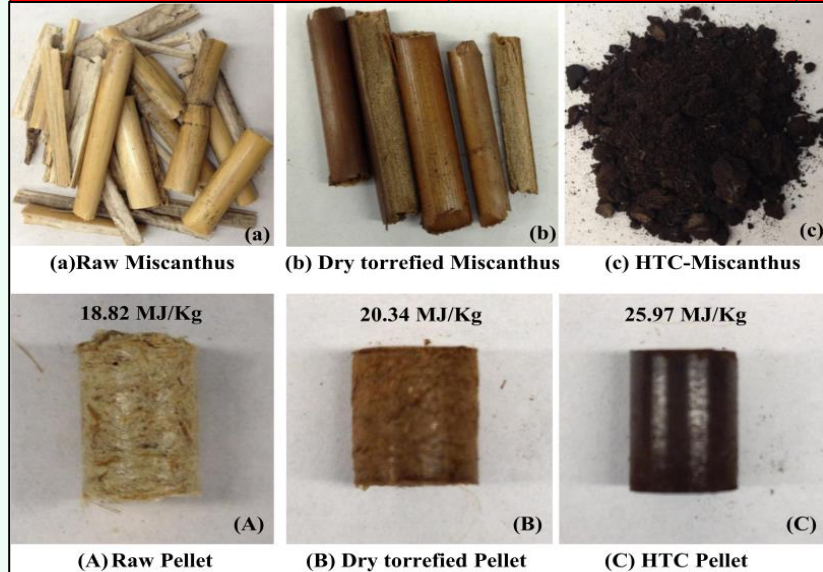


Significance:

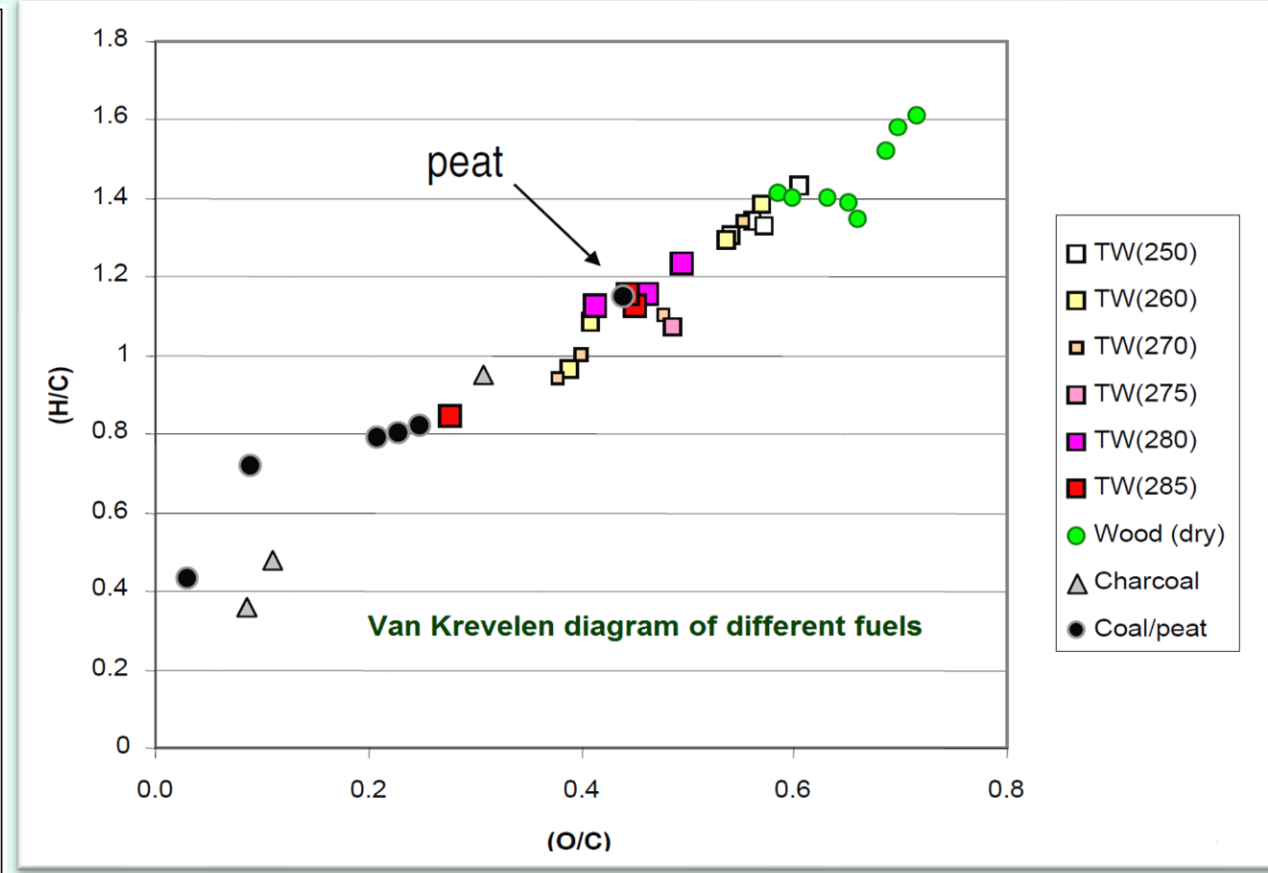
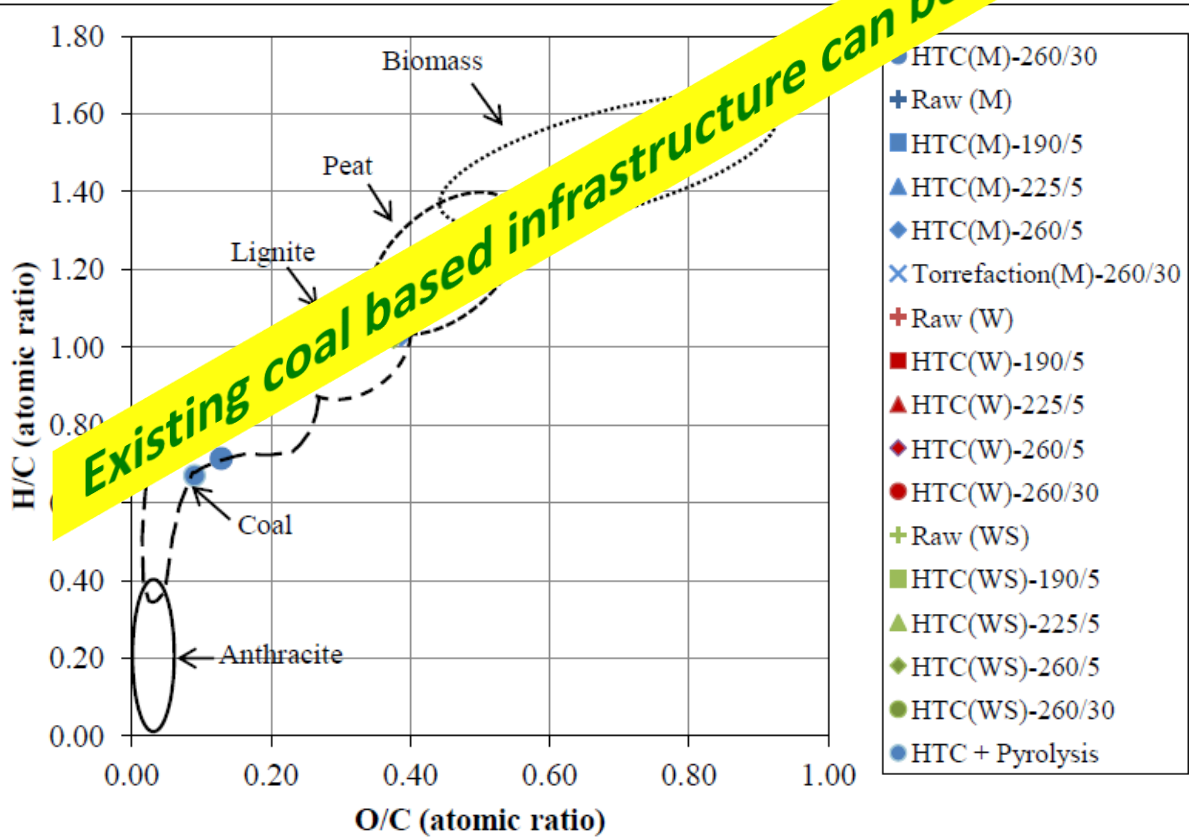
- HTC can be an ideal pre-treatment method to remove some of the barriers.
- Depending upon applications, it may require further processing

Effect on Mass and Energy Density

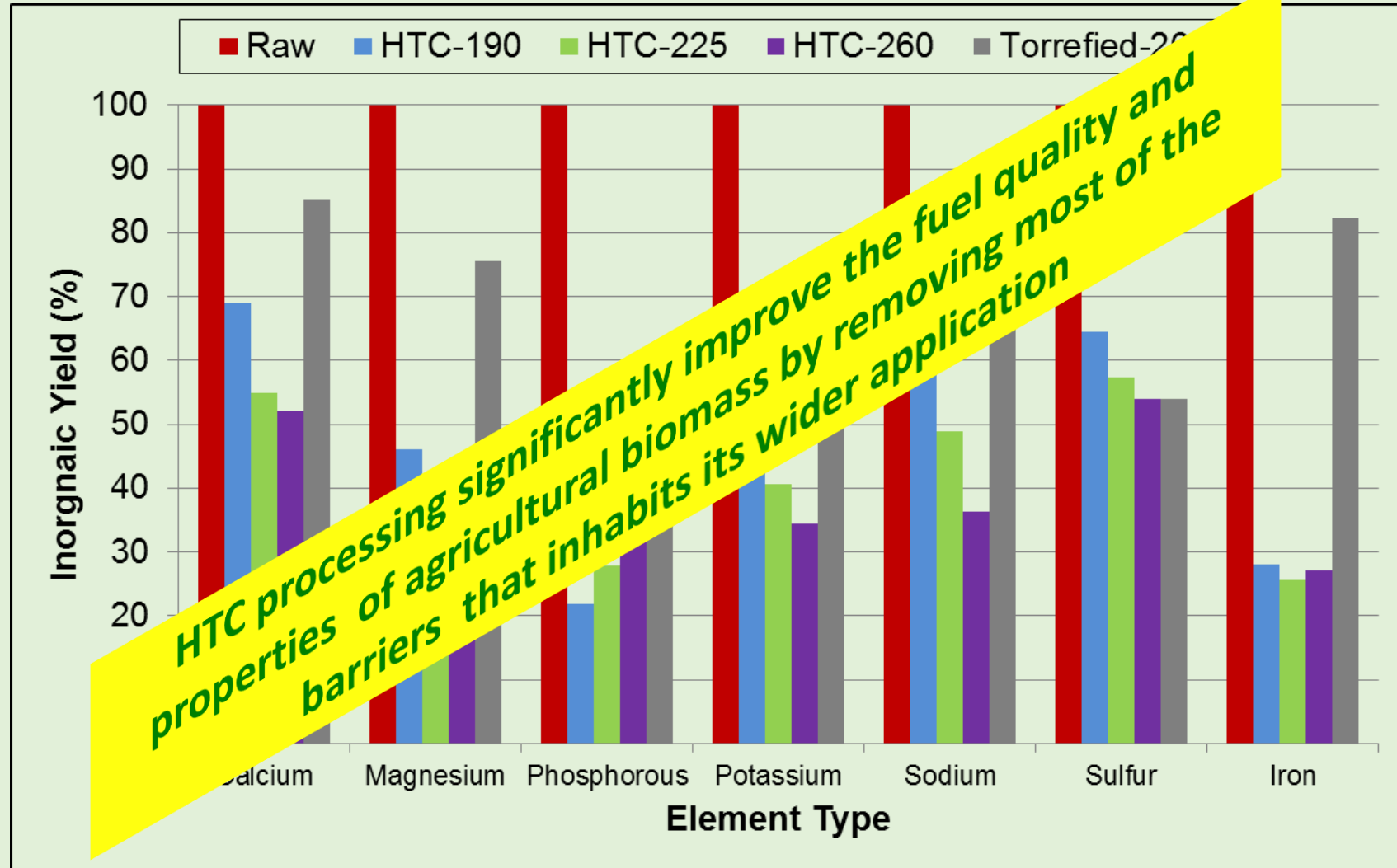
Material	Reaction Time (mins)	Mass Density (kg/m ³)	HHV (MJ/kg)	Energy Density (GJ/m ³)
Raw Biomass		321.09	18.47	5.93
Raw Pellet		834.05	18.82	15.69
HTC-190	5	886.87	20.19	17.9
HTC-225	5	959.39	21.62	20.74
HTC-260	5	1035.99	25.97	26.9
HTC-260	30	-	29.52	-
Torrefaction-260C	30	819.55	20.34	16.66



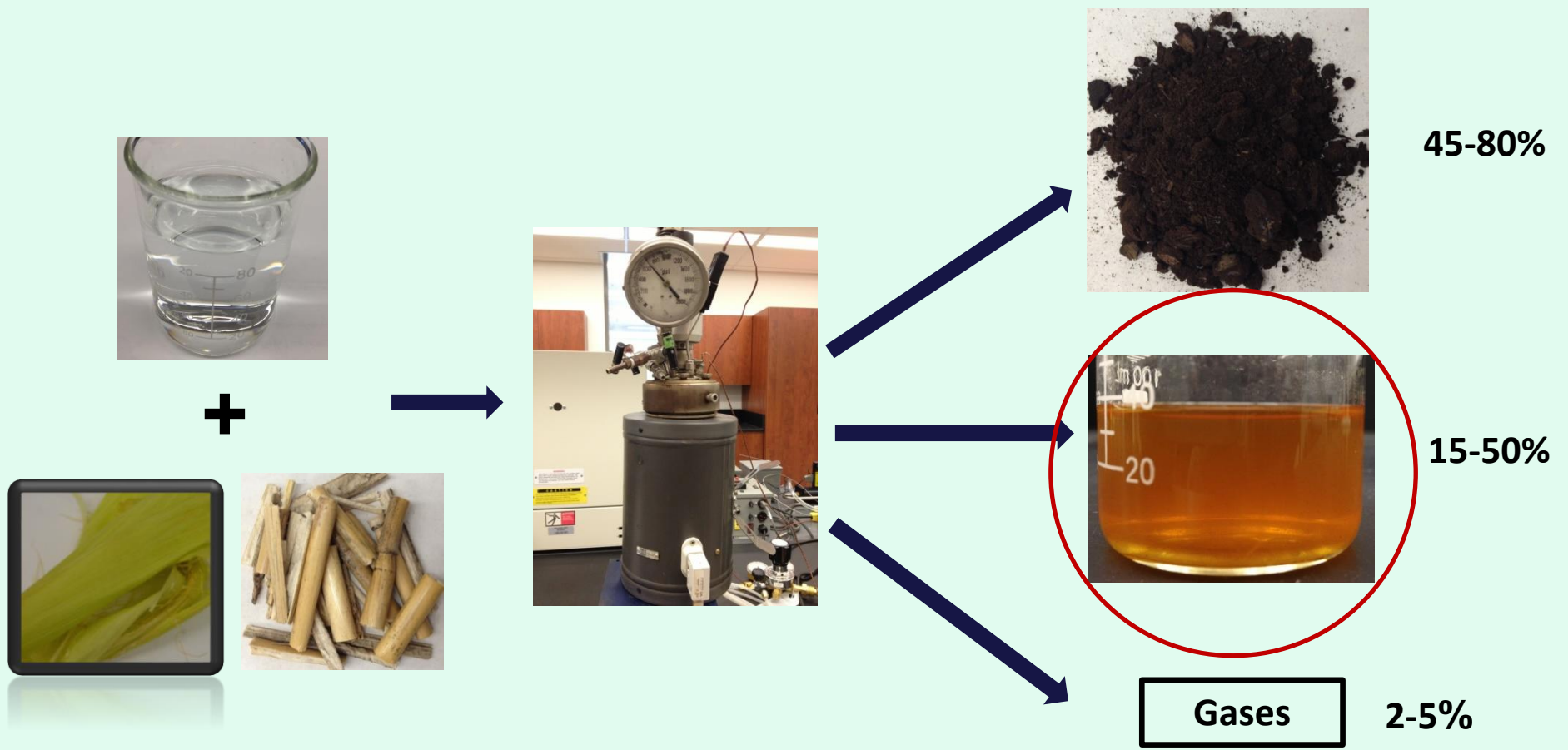
Van Krevelen diagram (Effect on H/C-O/C Atomic Ratios): Comparison of HTC and conventional torrefaction



Effect of Pre-treatment on Alkali and Alkaline Content



HTC Product Streams



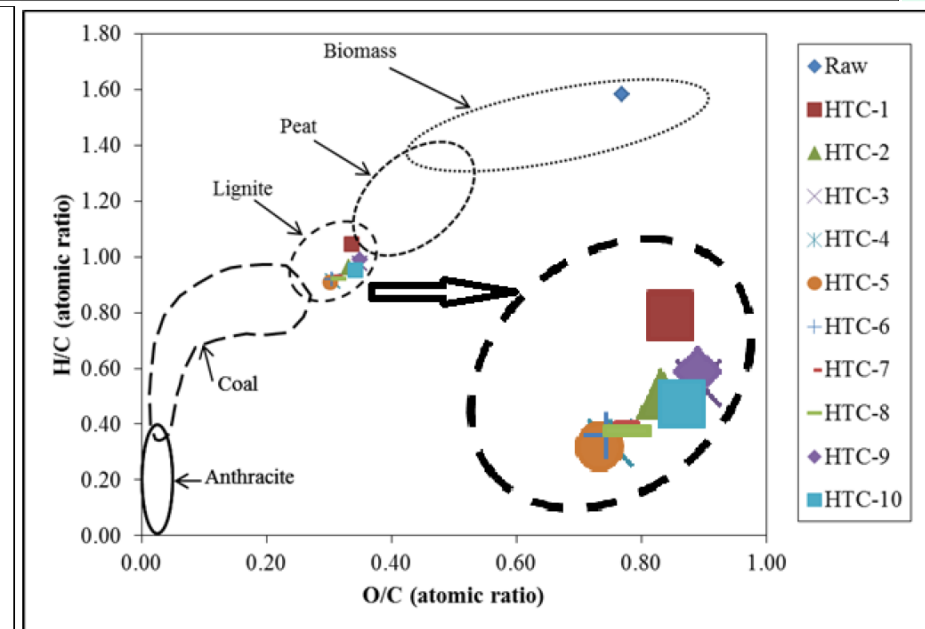
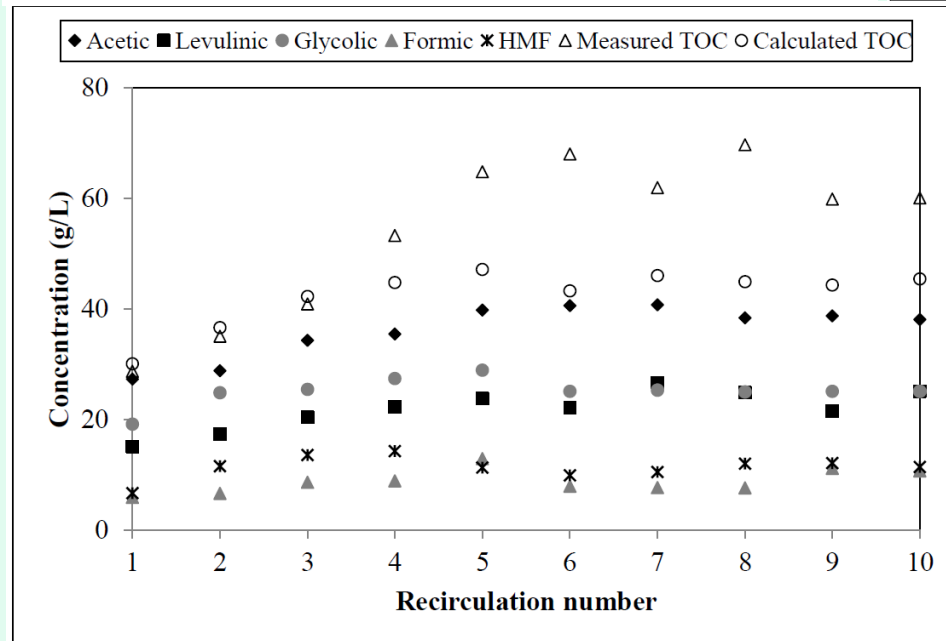
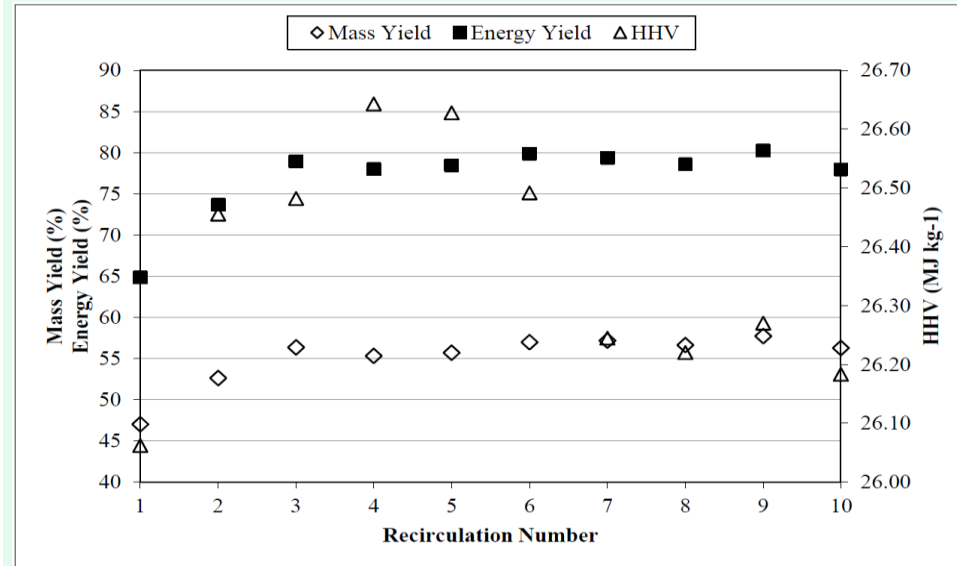
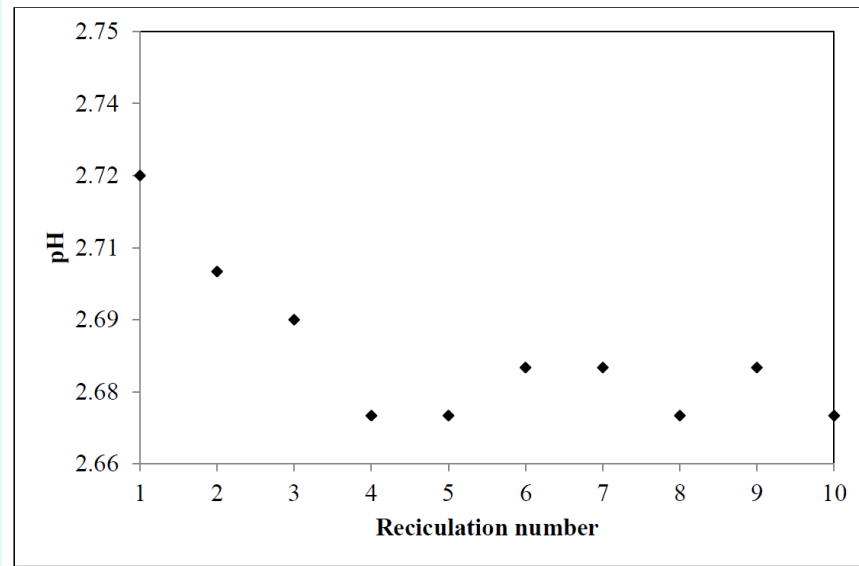
The distribution & properties of products strongly depend on reaction conditions!

Process water from the hydrothermal carbonization of biomass: a waste or a valuable product?

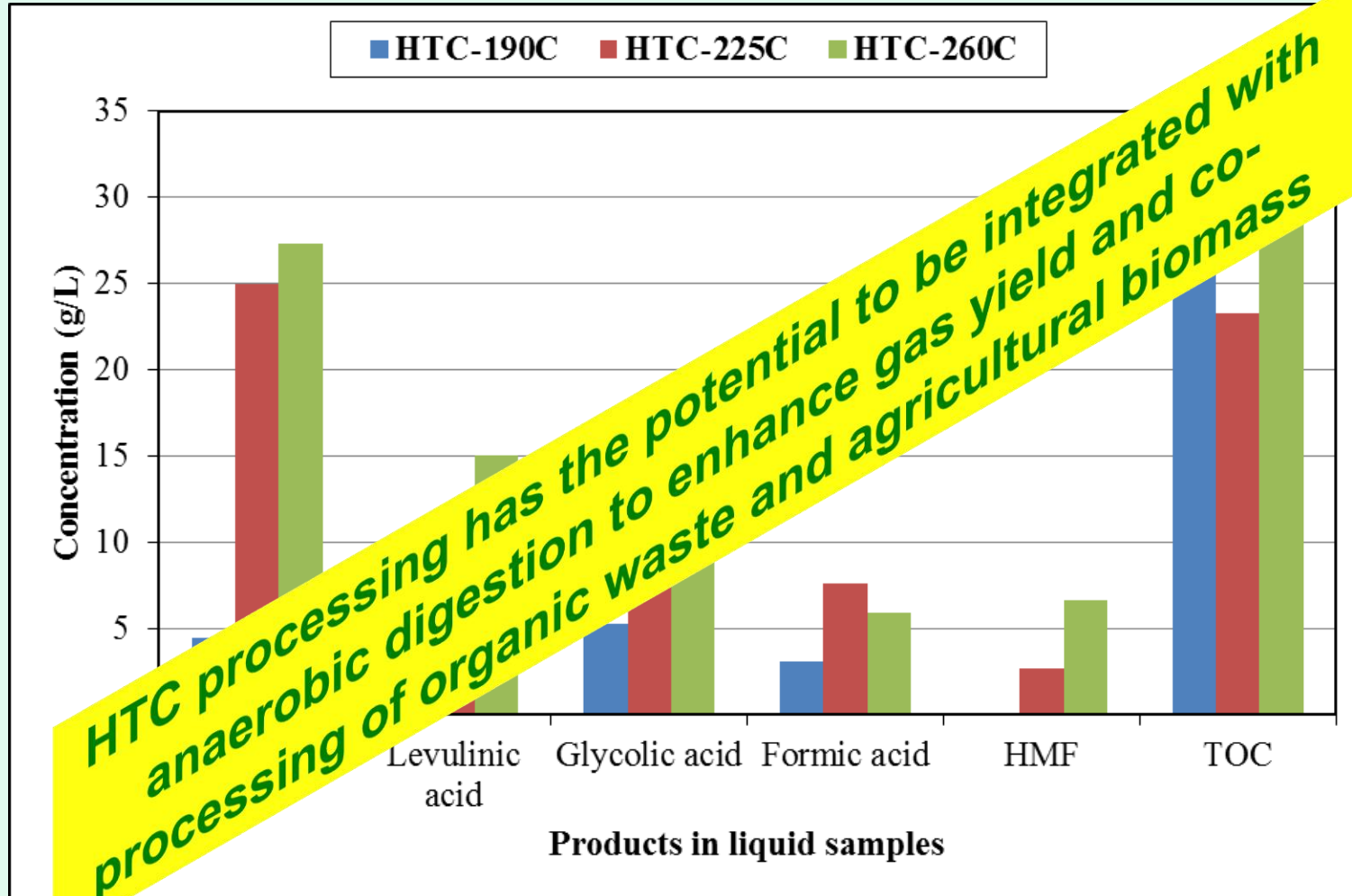
Waste Biomass Valor, 1-9, 2017



Recycling of Process water



Characterization of HTC Process Water



Example 1

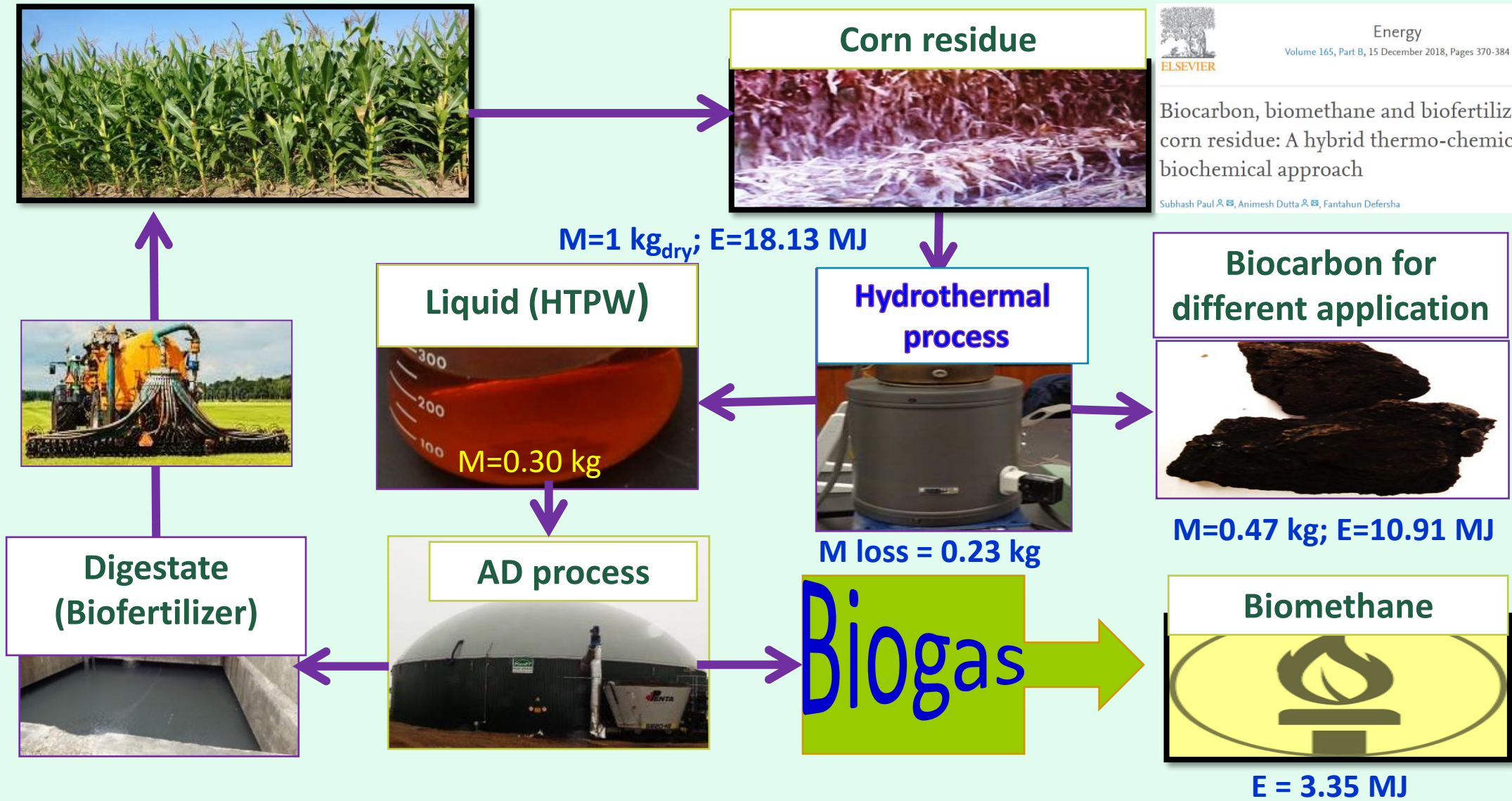
Bioenergy and biofertilizer from hydrothermal treated corn residue: a circular economy concept

Example 1: Bioenergy and biofertilizer from hydrothermal treated corn residue: a circular economy concept



Biocarbon, biomethane and biofertilizer from corn residue: A hybrid thermo-chemical and biochemical approach
 Subhash Paul, Animesh Dutta, Fantahun Defersha

Recovery N=31%,
 P=23%, K=26%, S=19%



Significance: Overall energy recovery efficiency=79%.

CASE STUDY 1: NUMERICAL ANALYSIS OF AN INTEGRATED HTC-AD SYSTEM FOR POWER GENERATION

Numerical Comparison of a Combined Hydrothermal Carbonization and Anaerobic Digestion System with Direct Combustion of Biomass for Power Production

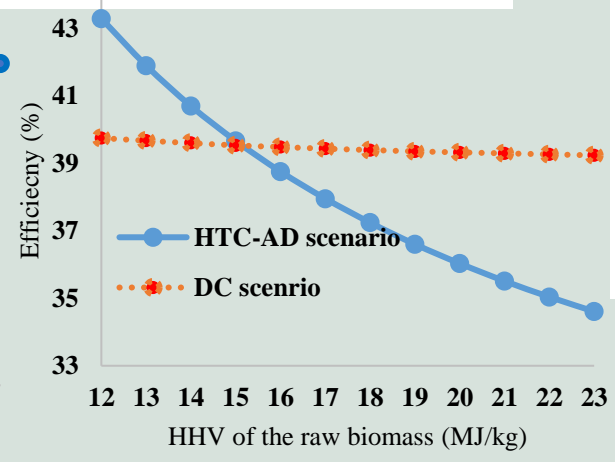
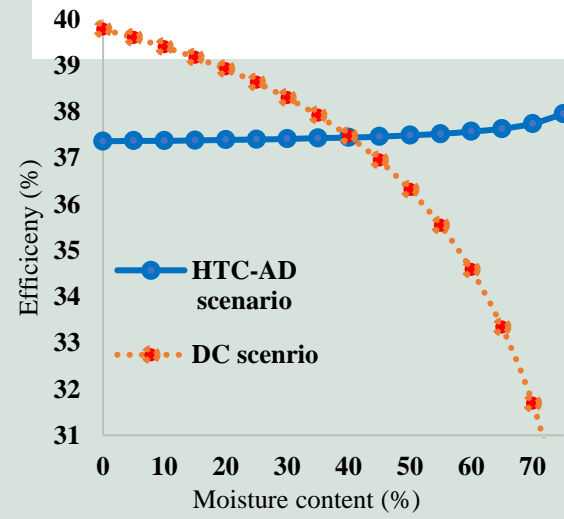
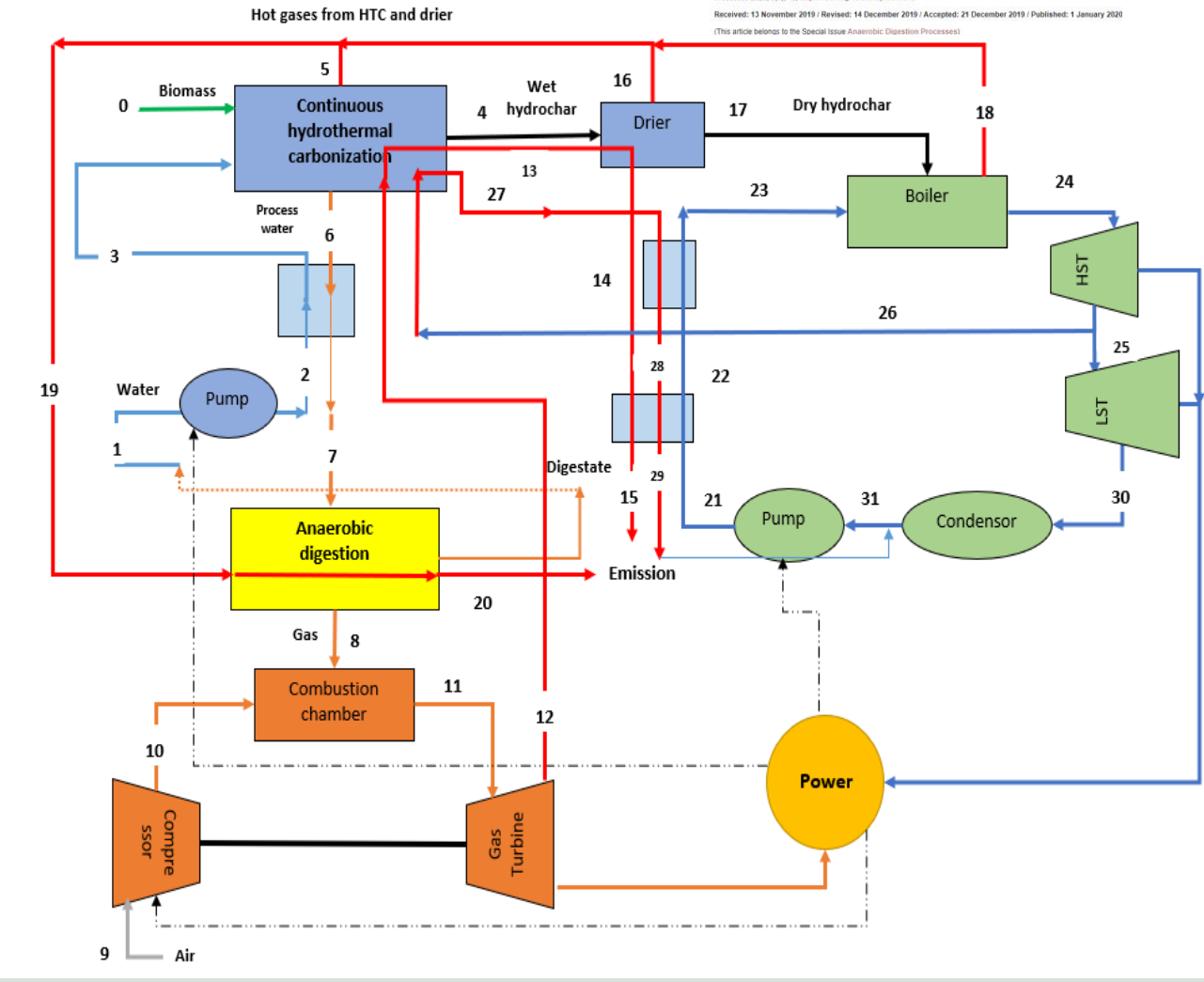
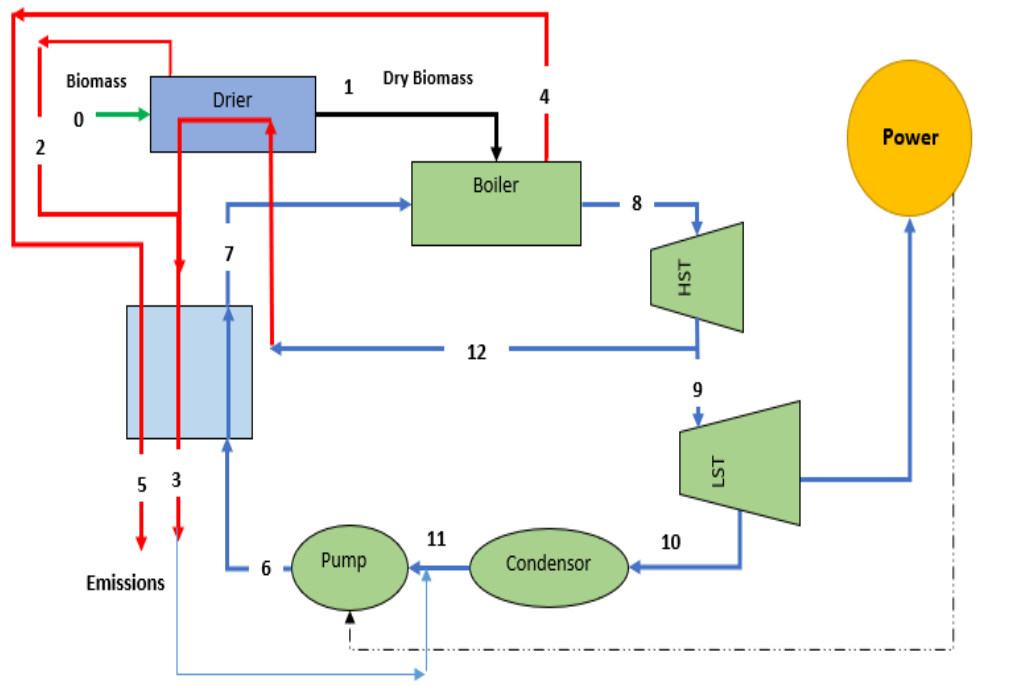
by [Muhammad Heidan](#)¹, [Shakirudeen Sabaudeen](#)¹, [Omid Norouzi](#)¹, [Bishnu Acharya](#)^{1,2} and [Azamshah Duttia](#)^{1*}

¹ School of Engineering, University of Guelph, Guelph, ON N1G 2W1, Canada
² School of Sustainable Design Engineering, University of Prince Edward Island, Charlottetown, PE C1A 4P3, Canada
 * Author to whom correspondence should be addressed.

Processes 2020, 8(1), 43; <https://doi.org/10.3390/pr8010043>
 Received: 13 November 2019 / Revised: 14 December 2019 / Accepted: 21 December 2019 / Published: 1 January 2020
 (This article belongs to the Special Issue Anaerobic Digestion Processes)



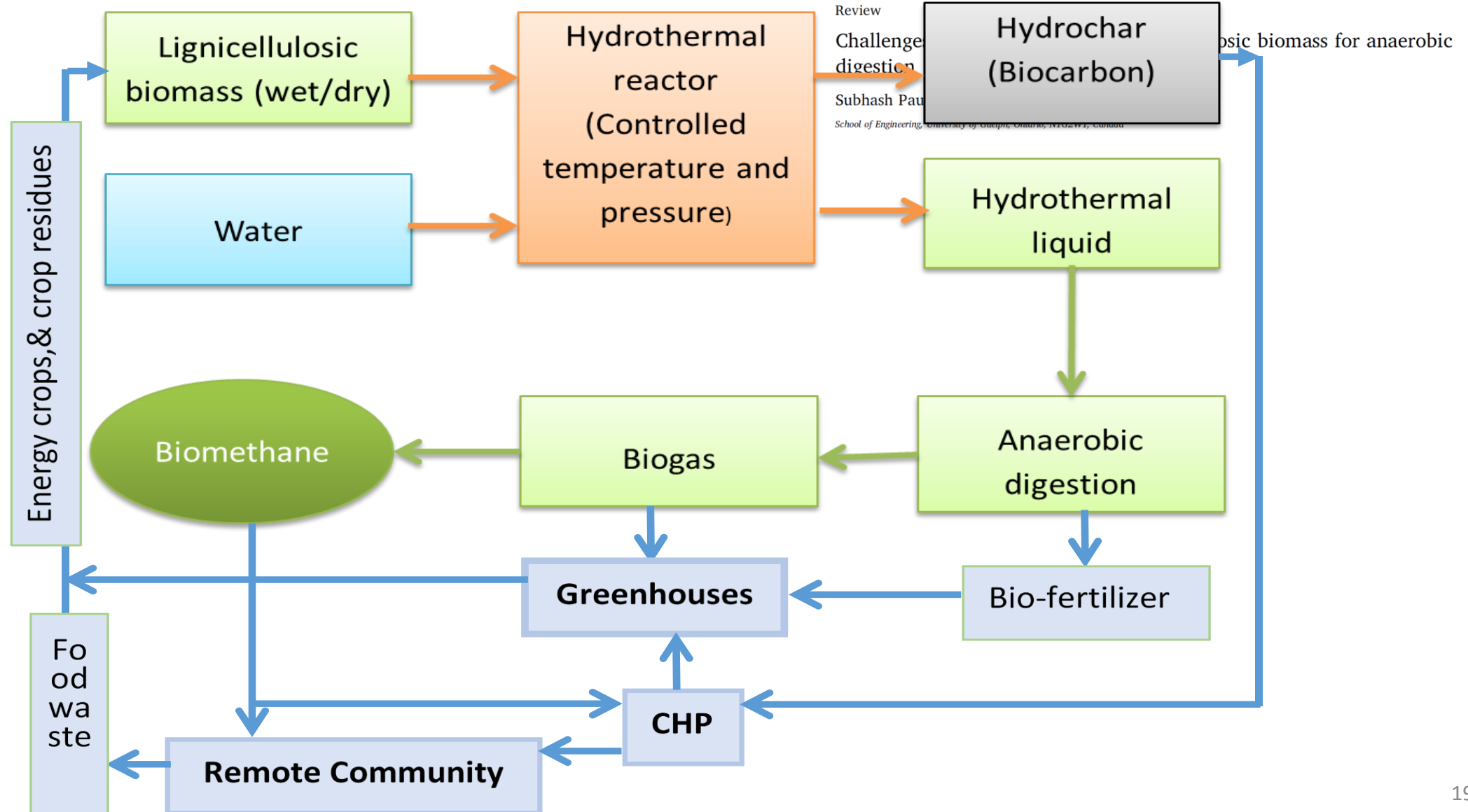
Ref: Proces...



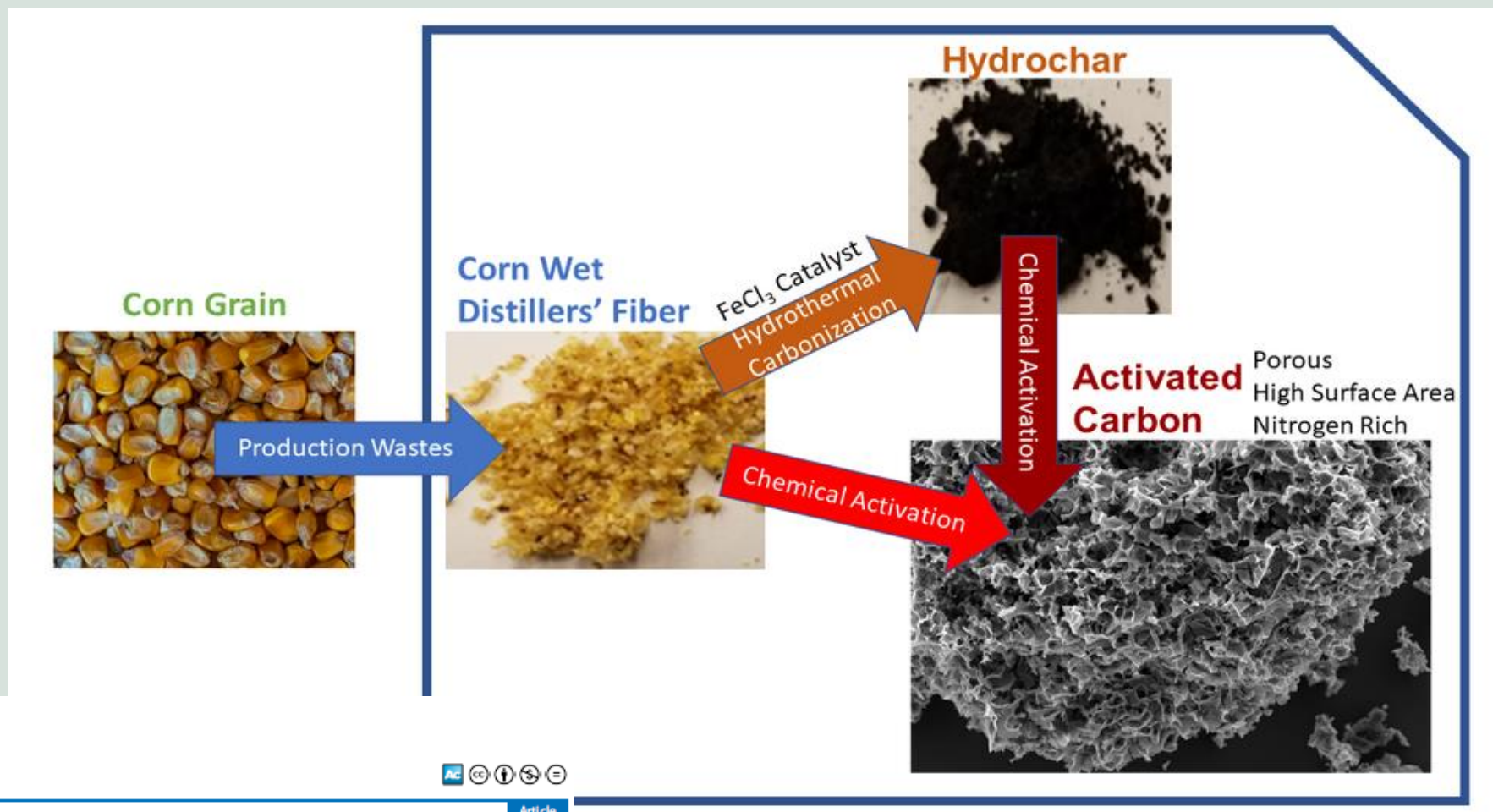
Significance: HTC-AD scenario shows a better performance compared to DC one when the moisture content of the biomass is over 40%



Review
Challenge
digestion
Subhash Paul
School of Engineering, University of Guelph, Ontario, N1G 2W1, Canada

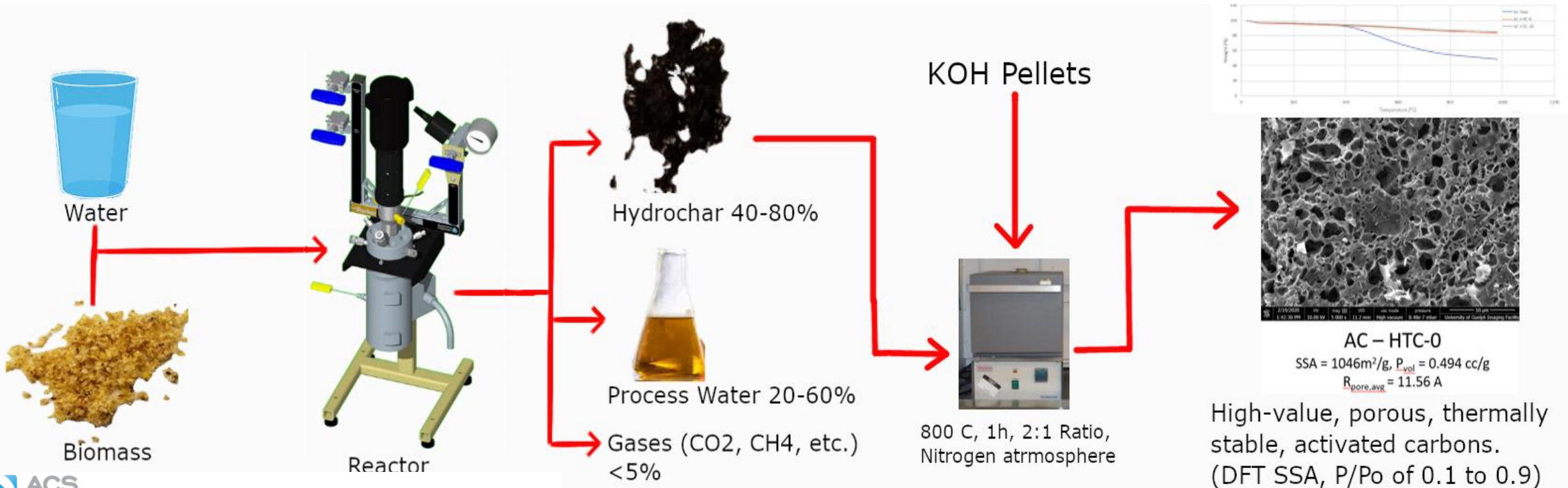


EX. 2: A TUNABLE APPROACH FOR ACTIVATED CARBON PRODUCTION FROM LOW VALUE BIOMASS



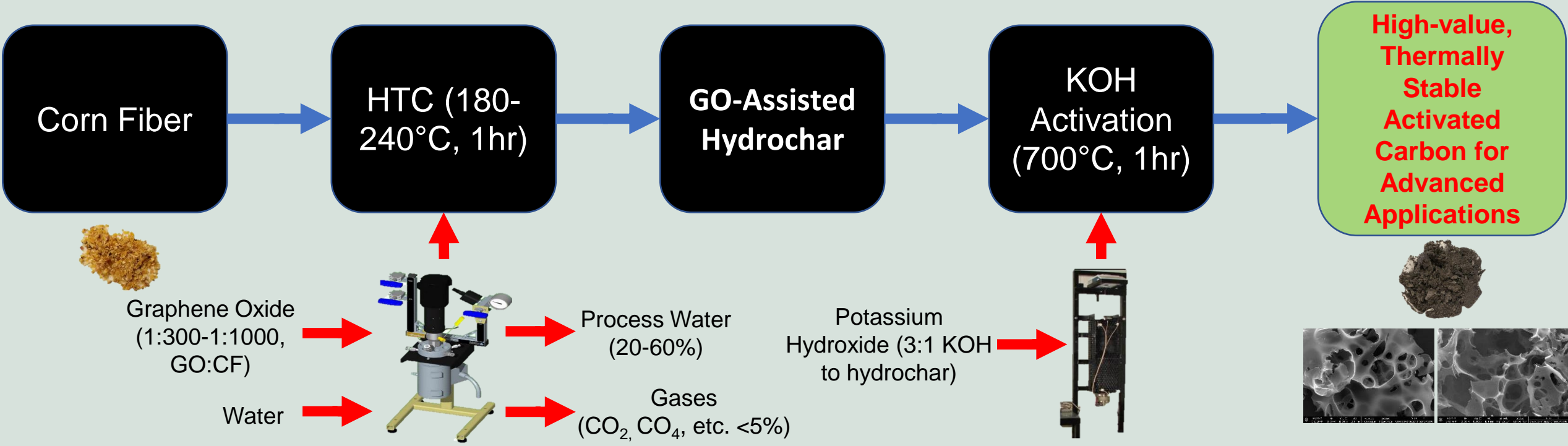
Significance:

- Valuable, high quality activated carbons can be produced through a 2-step HTC and chemical activation procedure.
- Applications in heavy metal removal, water filtration, gas storage, super capacitors, and many more.



PROPOSED NOVEL PROCESS

Proposed 2-Stage Process: GO Assisted HTC of Corn Fiber With KOH Activation



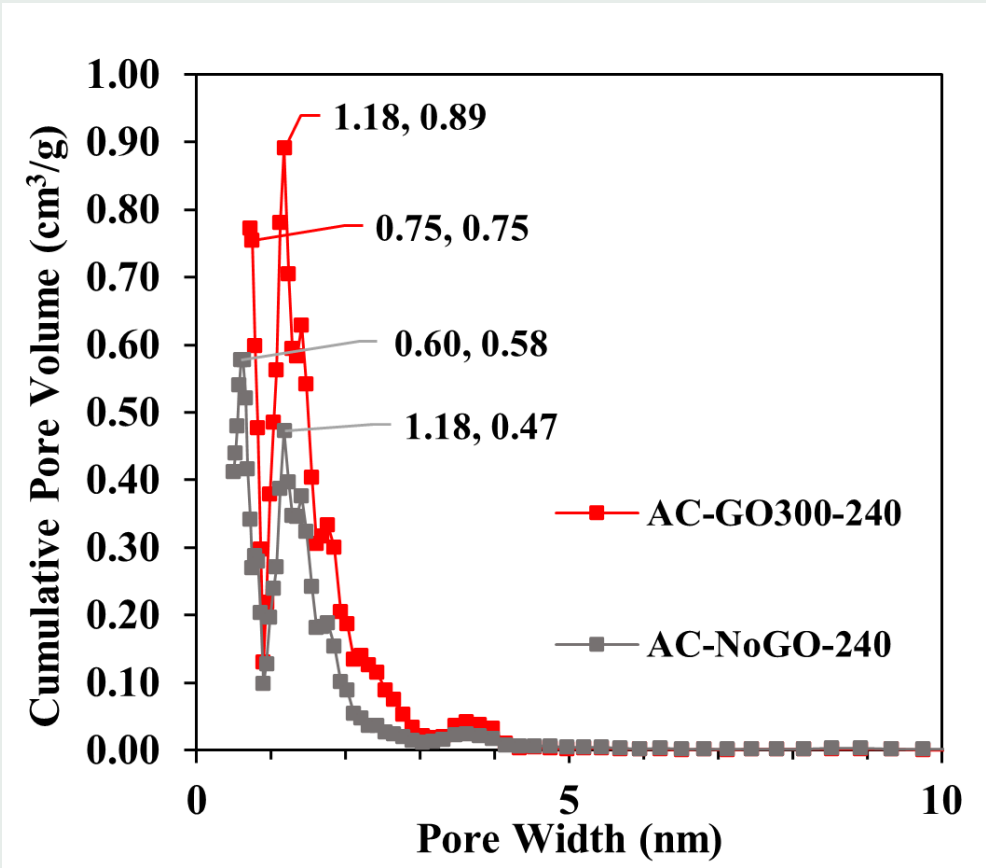
 Corn fiber is high in moisture and nitrogen content¹: Low-value by-product of corn ethanol industry ¹ Suitable for HTC, enhancement of H ₂ storage properties from N ₂ functionality ²	 Addition of GO (C₁₄₀H₄₂O₂₀): Act as catalyst during HTC, enhance morphology of hydrochar ^{3,4} Promote HTC reactions and facilitate carbonization ^{3,4}	 HTC for carbonization step Enhances pore formation and degree of carbonization ^{5,6} Consumes less energy than conventional carbonization methods ⁶ Reduction in ash content and impurities ⁶	 Chemical Activation with KOH Promote formation of micropores ⁷ Higher surface area ⁷
			22

RESULTS

	Nomenclature	BET Specific Surface Area (m ² /g)	Total Pore Volume (cm ³ /g)	Micropore Volume (cm ³ /g)
Hydrochar	NoGO-240	1.52	-	-
	GO1000-240	1.59	-	-
	GO800-240	3.43	-	-
	GO300-240	5.89	-	-
AC	AC-NoGO-240	1942.71	0.875	0.383
	AC-GO1000-240	2253.72	0.999	0.668
	AC-GO900-240	1866.16	0.829	0.558
	AC-GO800-240	1960.36	0.899	0.451
	AC-GO700-240	2023.19	0.912	0.477
	AC-GO600-240	1908.94	0.888	0.420
	AC-GO500-240	2144.96	0.989	0.489
	AC-GO400-240	1952.76	0.979	0.233
	AC-GO300-240	2549.10	1.098	0.486

RESULTS

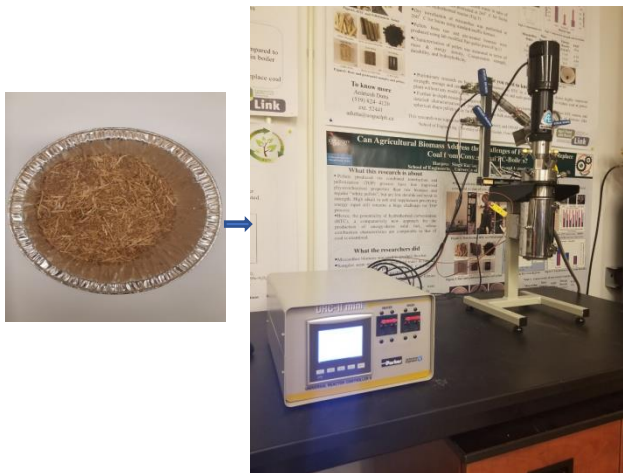
Nomenclature	Micropore Volume (cm ³ /g)
AC-NoGO-240	0.383
AC-GO1000-240	0.668
AC-GO900-240	0.558
AC-GO800-240	0.451
AC-GO700-240	0.477
AC-GO600-240	0.420
AC-GO500-240	0.489
AC-GO400-240	0.233
AC-GO300-240	0.486



- Increased micropore volume indicates added stability, resistance to pore enlargement from KOH activation

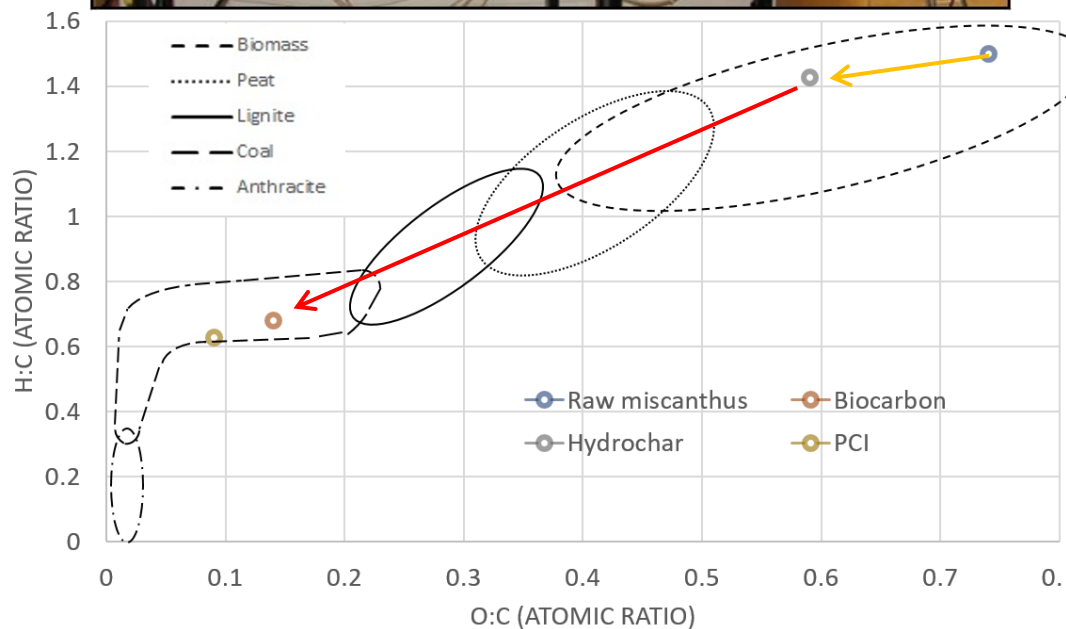
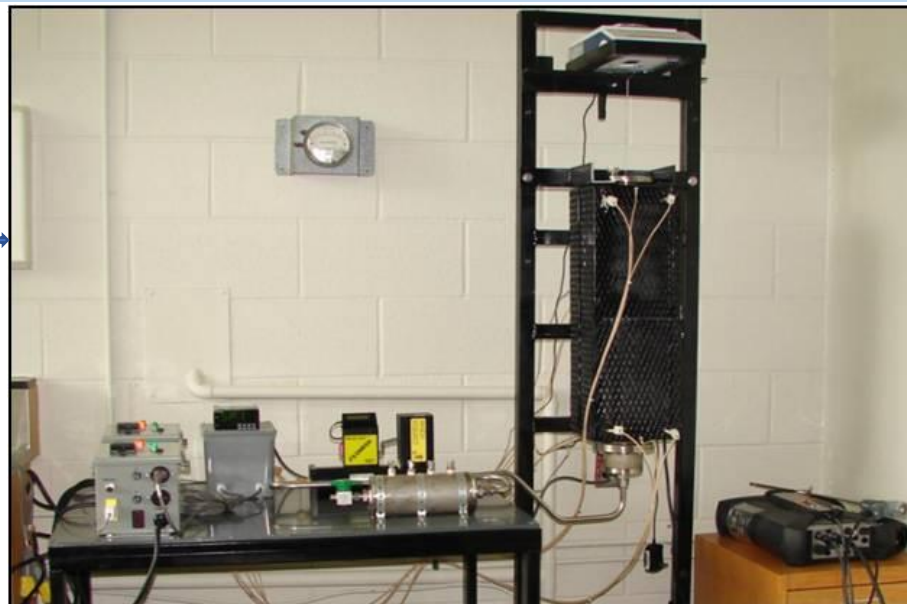
Hydrogen Storage Potential

- Most effective in micropores (0.7nm)
- Requires high surface area
- Presence of surface functional groups
- N-doping without extra step



Hydrochar	
C(%)	52.2
H(%)	6.2
N(%)	0.05
S(%)	0
O(%)	41.31
Ash(%)	0.24
FC(%)	15.1
VM(%)	84.66
HHV (MJ/Kg)	20.37

Properties	Raw Switchgrass	Torrefied-290
%C	44.76 ± 2.04	64.28 ± 2.42
%H	6.04 ± 0.62	4.34 ± 0.69
%N	0.66 ± 0.08	0.68 ± 0.13
%S	0	0
%O	44.09 ± 1.87	23.58 ± 1.87
HHV (MJ/Kg)	17.13 ± 1.49	26.04 ± 1.91
%VM	84.3 ± 3.18	50.35 ± 2.72
%Ash	4.45 ± 0.23	7.12 ± 0.38
%FC	11.25 ± 0.8	42.53 ± 1.83



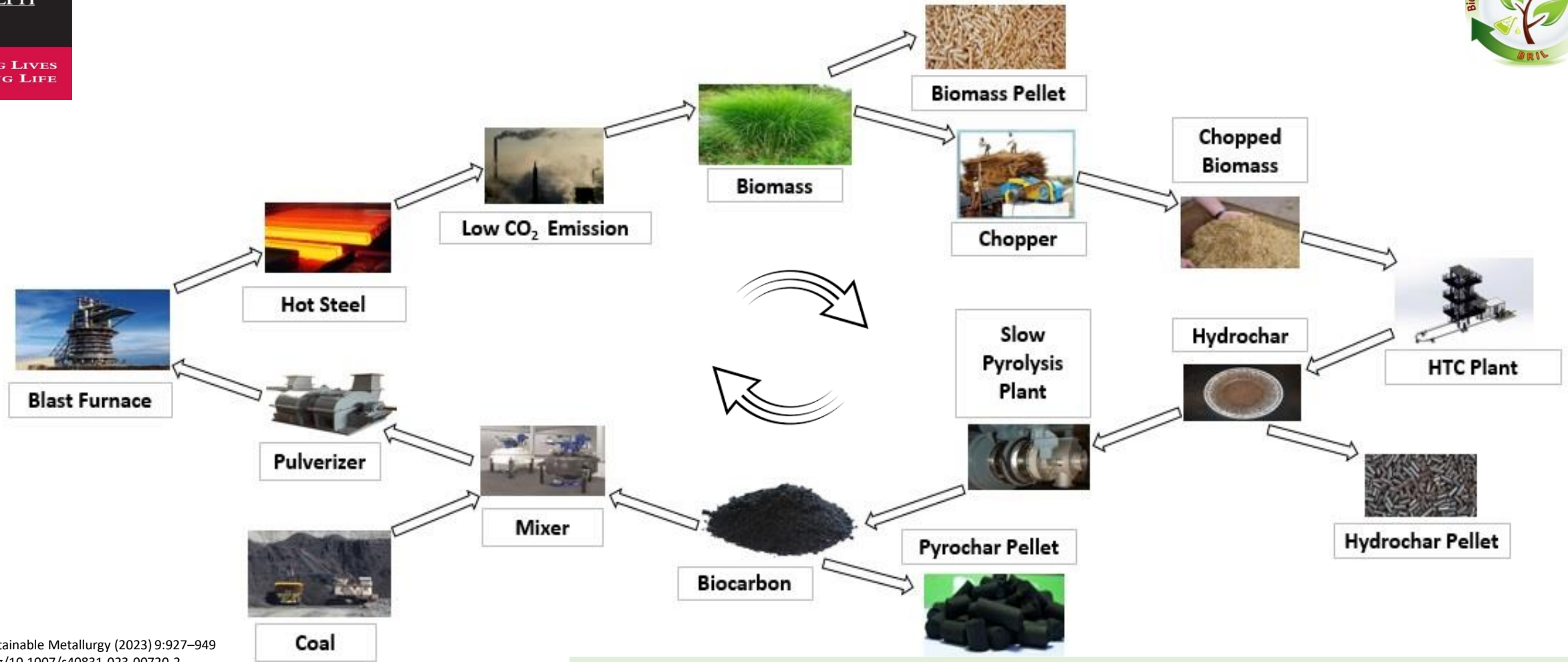
Biocarbon	
C(%)	79.67
H(%)	4.5
N(%)	0.35
S(%)	0
O(%)	14.69
Ash(%)	0.79
FC(%)	63.71
VM(%)	35.5
HHV (MJ/Kg)	32.59



PCI coal	
C(%)	77.66
H(%)	4.1
N(%)	1.76
S(%)	0.3
O(%)	9.53
Ash(%)	6.65
FC(%)	56.94
VM(%)	36.41
HHV (MJ/Kg)	32.07

Open Access Article
Miscanthus to Biocarbon for Canadian Iron and Steel Industries: An Innovative Approach
 by Trishan Deb Abhi, Omid Norouzi, Kevin Macdermid-Watts, Mohammad Heidari, Syeda Tasnim and Animesh Dutta*
 School of Engineering, University of Guelph, Guelph, ON N1G 2W1, Canada
 * Author to whom correspondence should be addressed.

Ex. 3 Continued: Overall Approach Industry relevance



Journal of Sustainable Metallurgy (2023) 9:927–949
<https://doi.org/10.1007/s40831-023-00720-2>
 REVIEW ARTICLE

Challenges and Opportunities of Agricultural Biomass as a Replacement for PCI Coal in the Ironmaking Blast Furnace:

A Review

Trishan Deb Abhi¹ · Kevin MacDermid-Watts¹ · Shakirudeen A. Salaudeen² · Aneela Hayder¹ · Ka Wing Ng³ · Ted Todoschuk⁴ · Animesh Dutta¹

Significance:

- Integrated HTC and slow pyrolysis of high ash low grade biomass
- Biocarbon with less ash content and good combustion behavior
- Partial replacement of fossil carbon in blast furnace iron making process
- Reduction of GHGs emission

Integrated hybrid architecture of metal and biochar for high performance asymmetric supercapacitors

Omid Norouzi, S. E. M. Pourhoseini, Hamid Reza Naderi, Francesco Di Maria & Animesh Dutta

Scientific Reports 11, Article number: 5387 (2021) | Cite this article

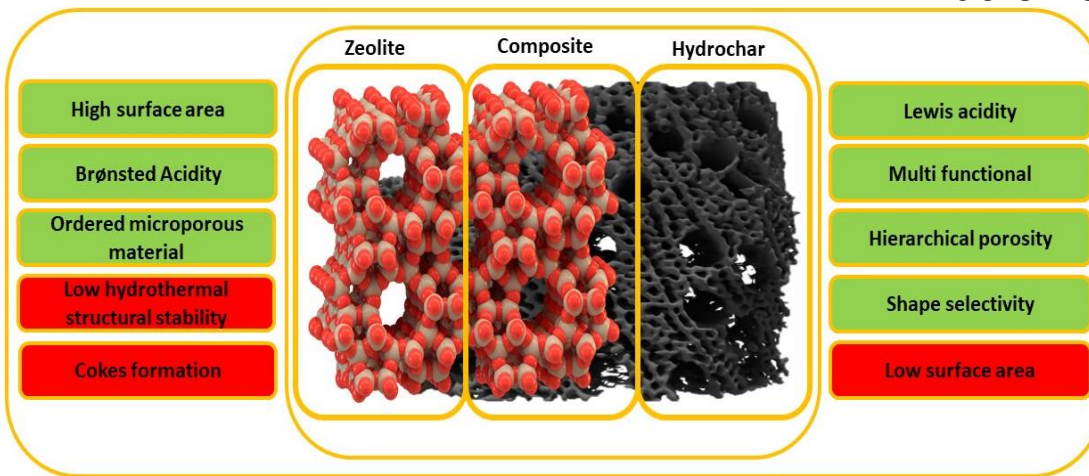
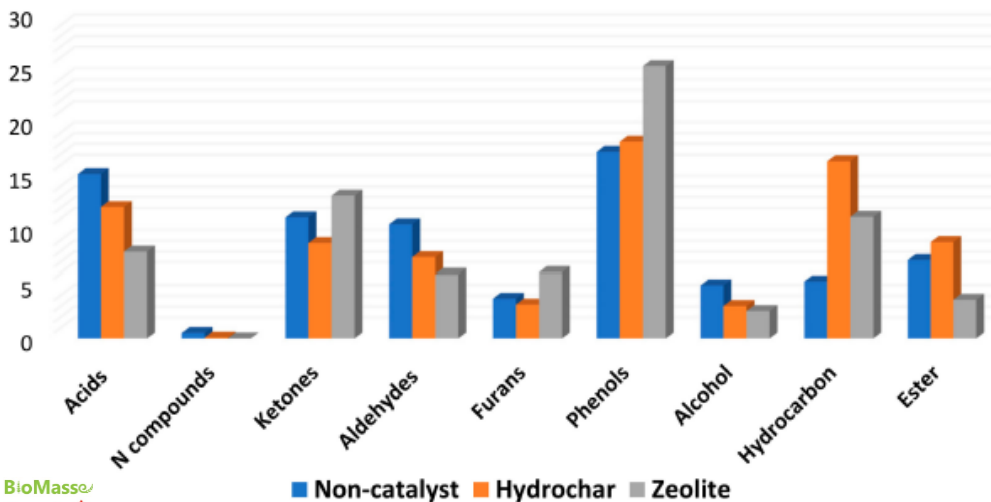
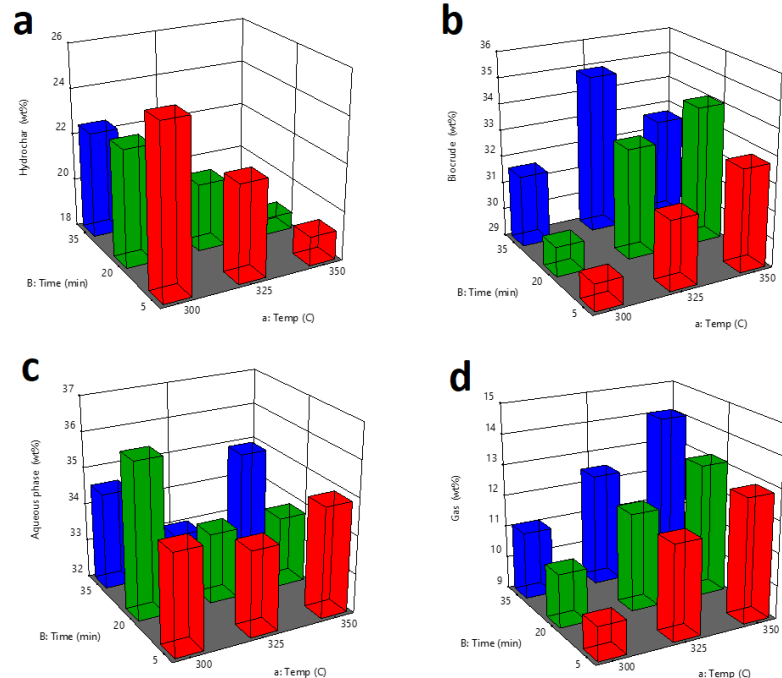
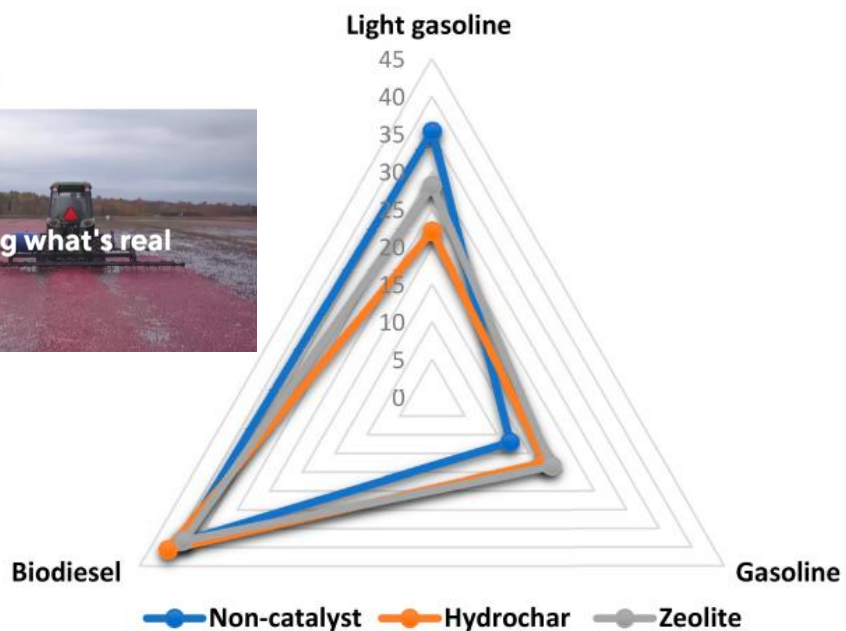
2220 Accesses | 12 Citations | 1 Altmetric | Metrics

A catalyst based on hydrochar and zeolite (hydrochar/zeolite composite) can resolve present limitations and challenges by:

- Creating meso/macropores into the micropores structure of the zeolite;
- Increasing the number of accessible active sites for macromolecules;

- Enhancing the thermal stability of the zeolite;
- Creating 3D interconnected structure using activated hydrochar

Case Study 4: New Insights for the Future Design of Composites Composed of Hydrochar and Zeolite from Cranberry Pomace. Ref: Energies 13 (24), 6600, 2020



Volume 412 15 May 2021 ISSN 1385-8947

ELSEVIER

CEJ CHEMICAL ENGINEERING JOURNAL

OZONATION OF SULFAMETHOXAZOLE
Ordered Mesoporous Alumina

Al₂O₃ Fe-doping (4.7 wt.% Fe)

X_{Toc} = 86%

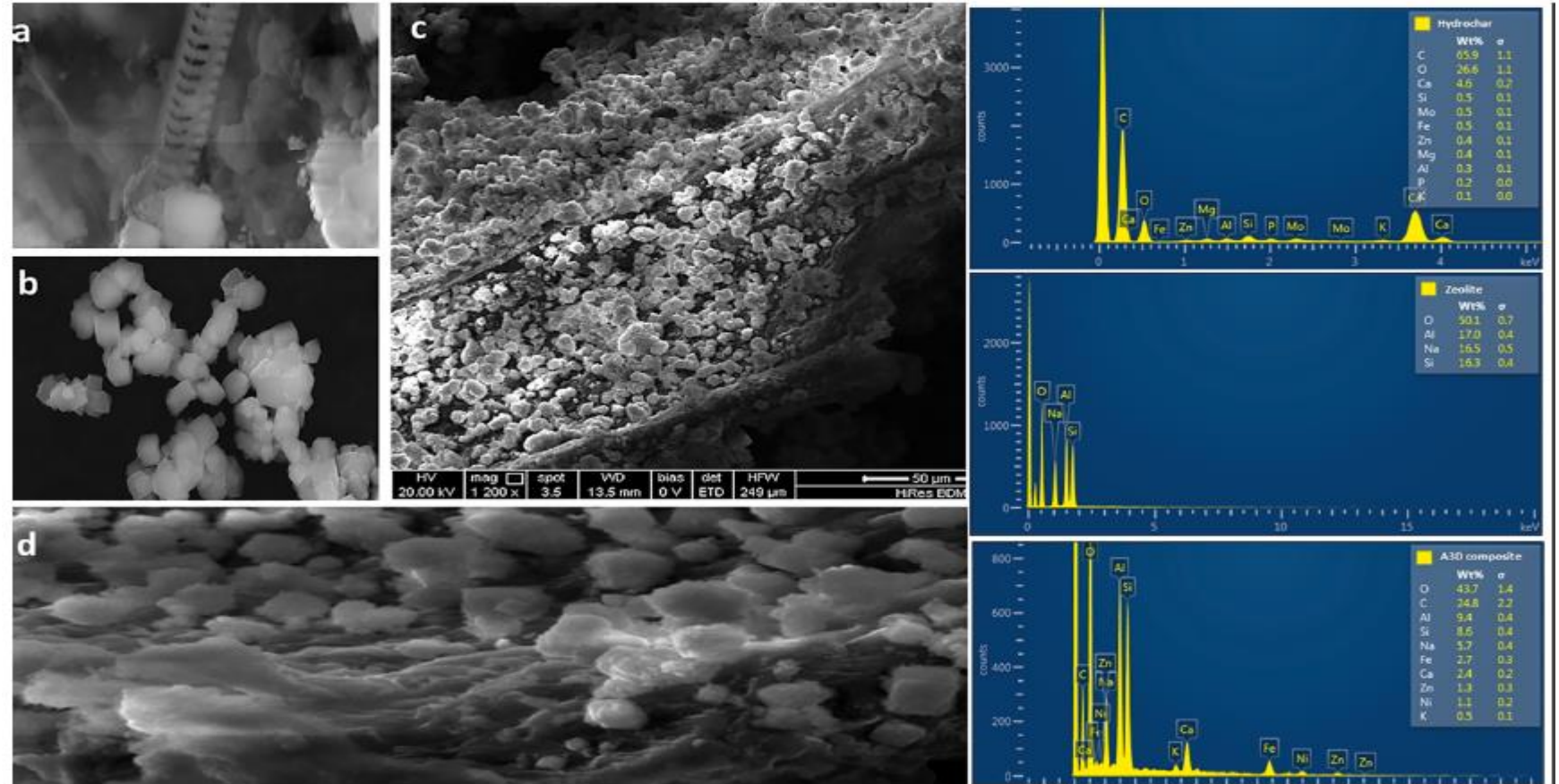
ADSORPTION removal
Poor catalytic contribution

EVAPORATION-INDUCED SELF-ASSEMBLY

Vol. 410, 15 April 2021, 128323

FEATURED ARTICLE
ENVIRONMENTAL CHEMICAL ENGINEERING
On disclosing the role of mesoporous alumina in the ozonation of sulfamethoxazole: Adsorption vs. Catalysis
Carla di Luca, Natalia Inchaurredo, Mireia Marcé, Rodrigo Parra, Santiago Espulgues, Patricia Haure

Wanqian Guo, Qi Zhao, Juanshan Du, Huazhe Wang, Xiaofan Li, Nanqi Ren



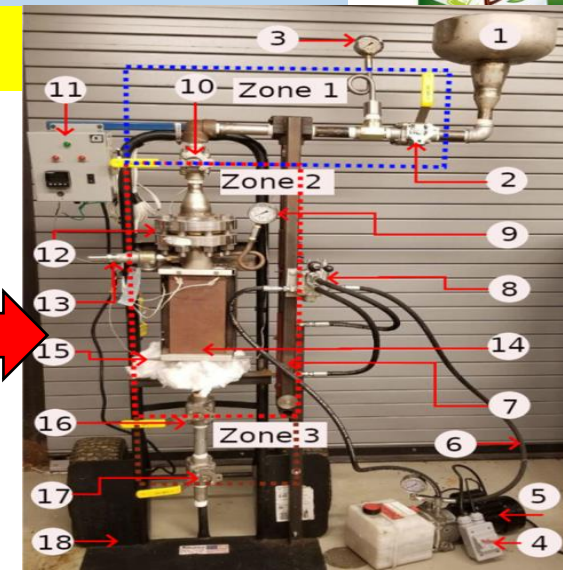
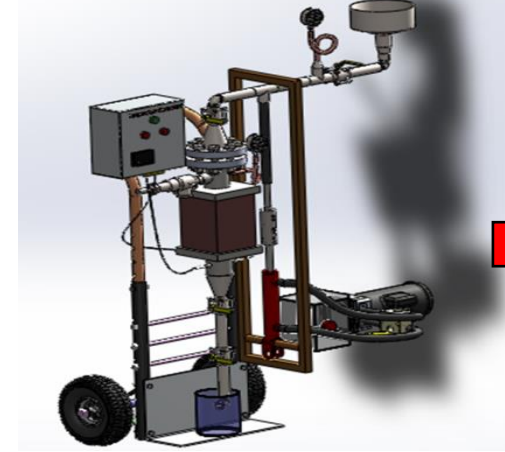
- We have synthesized a zeolite-hydrochar composite using a simple one-step hydrothermal liquefaction (HTL) process.
- hierarchically structured alumina porosity of the composite facilitates diffusion of macromolecules and their derivatives inside the composite and improves the accessibility to lewis acid sites.
- The chemical interaction of hydrochar/zeolite was confirmed by XRD and SEM-EDS analyses

Technical Progress

Lab scale continuous hydrothermal carbonization (HTC) reactor

A lab-scale continuous hydrothermal carbonization (HTC) reactor is developed and validated.

Conceptual Design

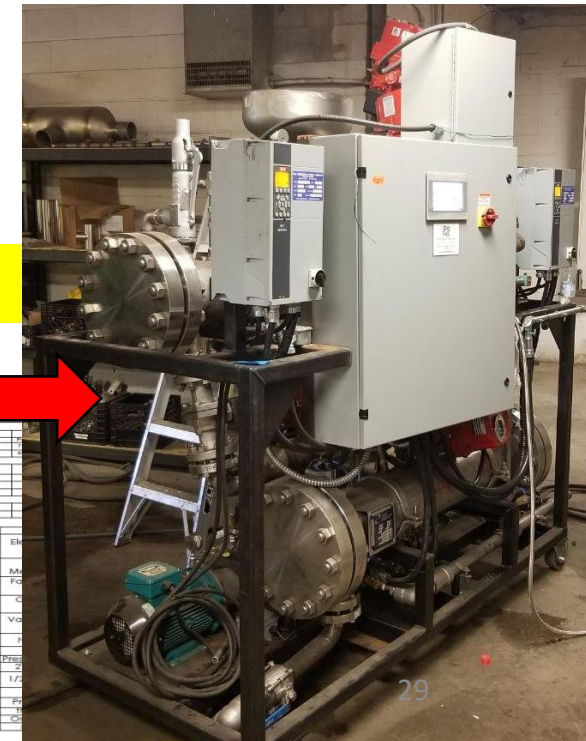
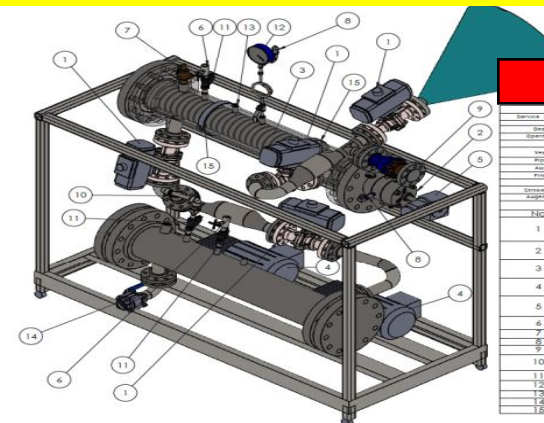


Continuous HTC is favorable due to the enhancement of primary carbonization and suppression of secondary carbonization; higher qualities of the hydrochar can be obtained.

Pilot scale continuous hydrothermal carbonization (HTC) reactor

A continuous pilot scale HTC reactor being developed. The process was validated with laboratory scale trials.

Conceptual Design





Opportunities for renewable hydrogen and
chemicals from waste resources

Funding from NSERC, AAFC and OMAFRA



Applied Catalysis B: Environmental 189 (2016) 119–132

Comparison of hydrogen yield and CGE to other researches

Study and experimental conditions	Catalytic condition	Hydrogen Yield (mol/mol _{carbon})	Carbon Gasification Efficiency
Present study Cattle manure biocrude (700 °C, 25 MPa)	Unanalyzed	0.50	0.89
	Ni10%- Ru0.08%/Al₂O₃-ZrO₂	1.10	0.95
Zhang et al. Sludge and waste newspaper biocrude (700 °C, 24 MPa)	Unanalyzed	0.52	0.90
	Ni10%- Ru0.1%/Al₂O₃	0.49	0.97
	Ni10%-Ru0.1%/AC	0.85	0.92
Byrd et al. Switchgrass biocrude (600 °C, 25 MPa)	Ru/TiO₂	0.81	0.78
	Ru/ZrO₂	--	0.67
	Ni/TiO₂	0.69	0.74
	Ni/ZrO₂	0.95	0.96
	Co/TiO₂	0.56	0.83
	Co/ZrO₂	0.71	1.02



Contents lists available at ScienceDirect

Applied Catalysis B: Environmental

journal homepage: www.elsevier.com/locate/apcatb



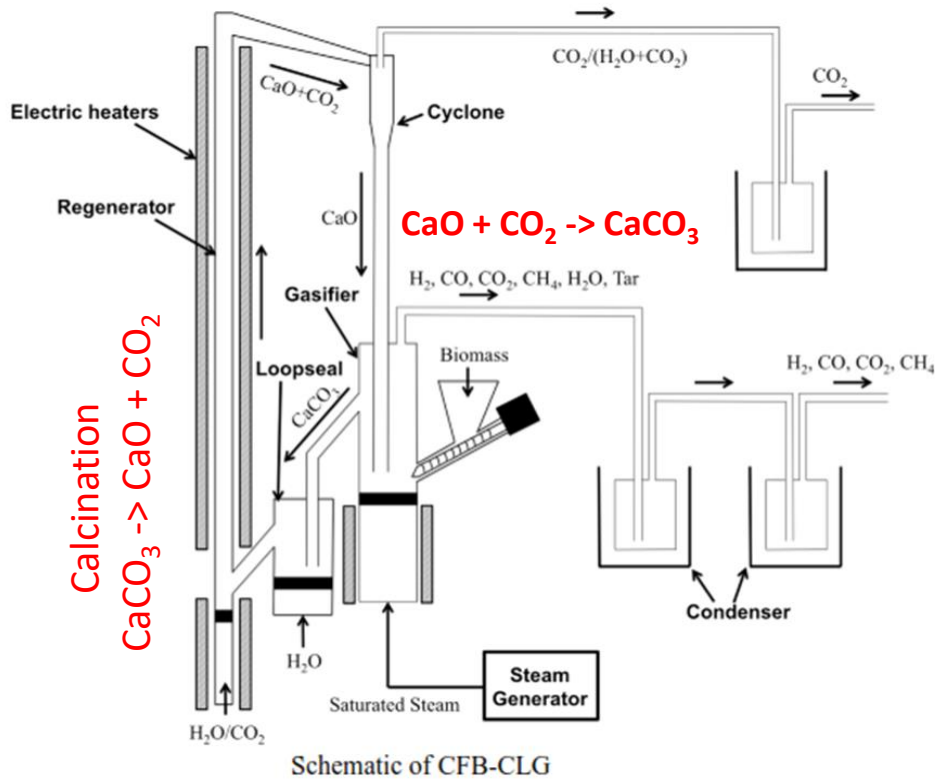
Catalytic supercritical gasification of biocrude from hydrothermal liquefaction of cattle manure

Mohammad S.H.K. Tushar^a, Animesh Dutta^{a,*}, Chunbao (Charles) Xu^b

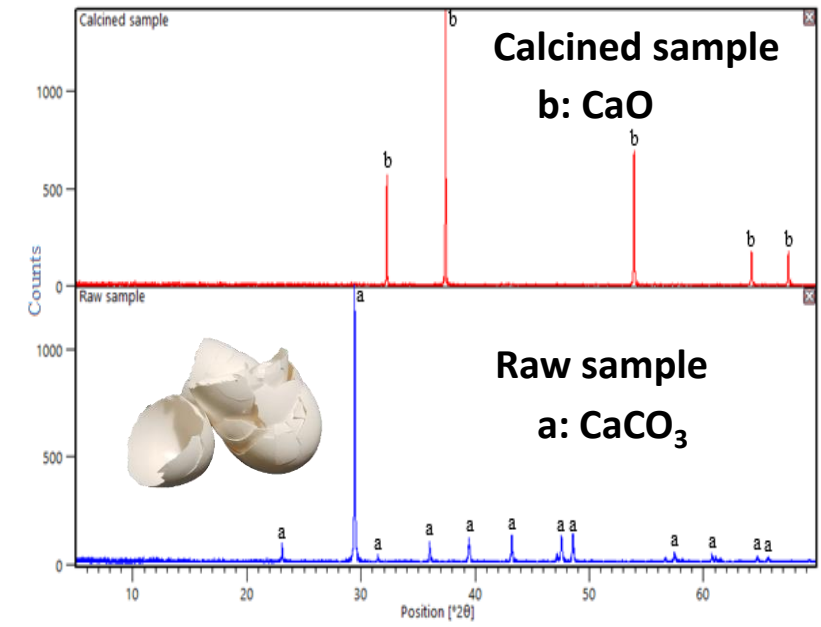
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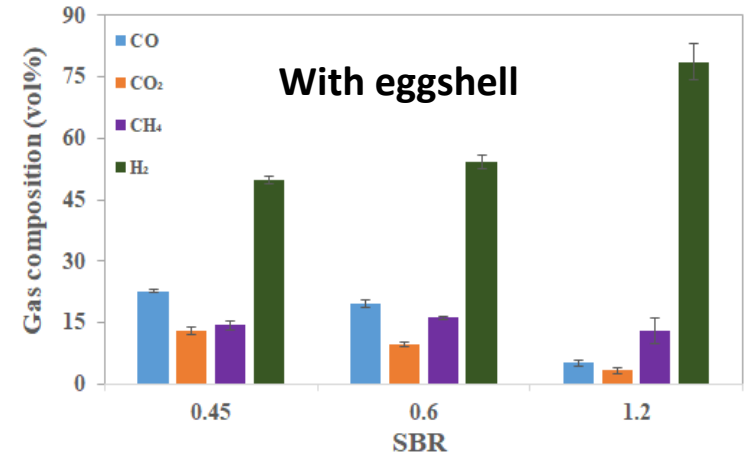
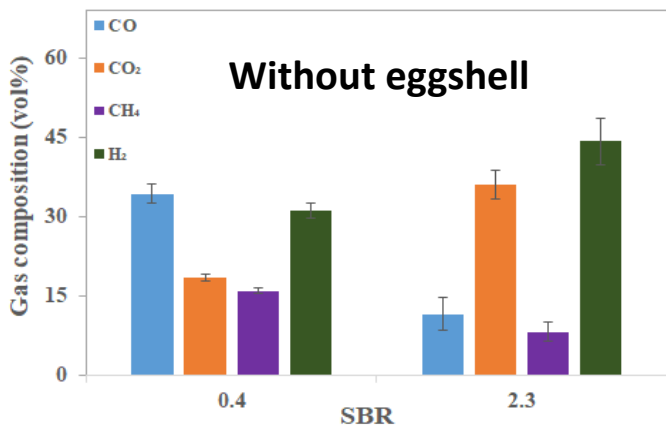
Hydrogen-rich gas stream from steam gasification of biomass: Eggshell as a CO₂ sorbent



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XRD results show that eggshell is mainly CaCO₃
Eggshell has been converted from CaCO₃ to CaO



- The utilization of eggshell in gasification has been experimentally investigated.
- The inclusion of calcined eggshell in the process reduced CO₂ concentration and increased hydrogen concentration

Pilot Plant System for Processing Waste

Brief system description

- The pilot plant system for the conversion of waste was recently patented by the United States Patent and Trademark Office (USPTO, US 11,033,869 B1, 2021)
- The system has both a fixed bed reactor and a fluidized bed reactor, which may be operated separately or simultaneously.



A picture of the patented pilot plant

(12) **United States Patent**
Al-Salem et al. (10) **Patent No.: US 11,033,869 B1**
(45) **Date of Patent: Jun. 15, 2021**

<p>(54) SYSTEM FOR PROCESSING WASTE</p> <p>(71) Applicant: KUWAIT INSTITUTE FOR SCIENTIFIC RESEARCH, Safat (KW)</p> <p>(72) Inventors: Sultan Al-Salem, Safat (KW); Animesh Dutta, Guelph (CA); Majed Hameed Al-Wadi, Safat (KW)</p>	<p>(56) References Cited</p> <p>U.S. PATENT DOCUMENTS</p> <p>5,853,548 A 12/1998 Piskorz et al. 8,100,990 B2 1/2012 Ellens et al. 10,550,330 B1 2/2020 Yelvington et al.</p> <p>(Continued)</p>
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Introduction (USDOE Project)

Plastic and Tire waste Management

Issues

- Over 90% of plastic waste and 65% of tire waste find their way to landfills
- Non-biodegradable in nature
- Disturb the land and marine environment



Opportunities

- High heating value of plastics (40-42 MJ/kg) and tires (28-32 MJ/kg)
- Commercial waste to energy technologies (such as pyrolysis) available in market



Analysis of Pyrolytic Wax from Plastics (HDPE)

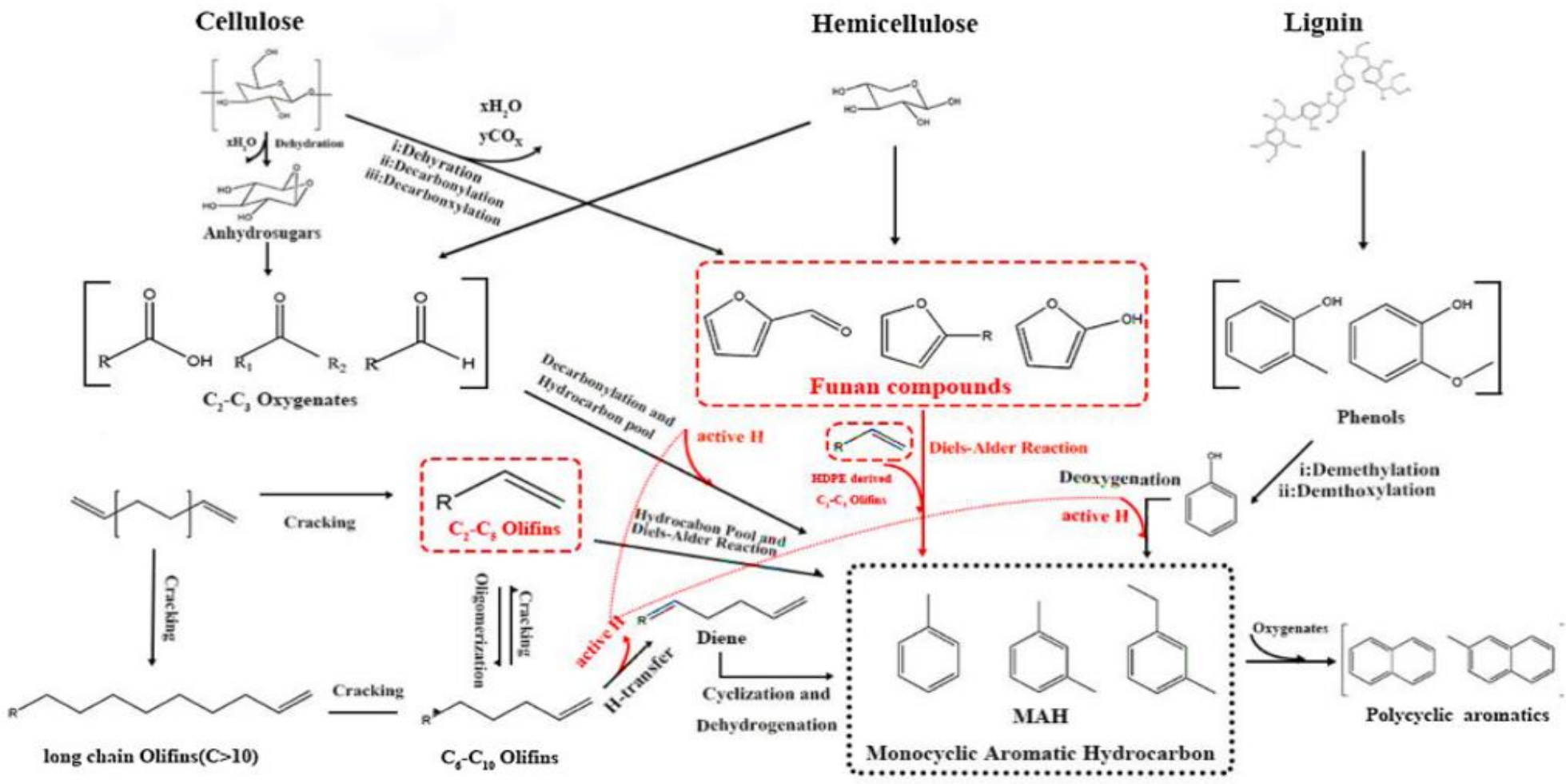


- The recovery of wax from the pyrolysis of high-density polyethylene in a patented pilot scale system has been investigated.
- Pyrolysis experiments were performed at 500 C in the fluidized bed section of the patented system.
- Characterizations of the feedstocks and obtained waxes were conducted with TGA-DSC, FTIR, GCMS, elemental analysis, and heating value.

Product distribution from the experiment

	Product distribution
Total feed (kg)	5.00
Wax (wt.%)	45.56
Oil (wt.%)	9.00
Char (wt.%)	0.10
Gas (wt.%)	45.34

Possible reaction pathway for aromatic hydrocarbons formation of co conversion of plastic and biomass





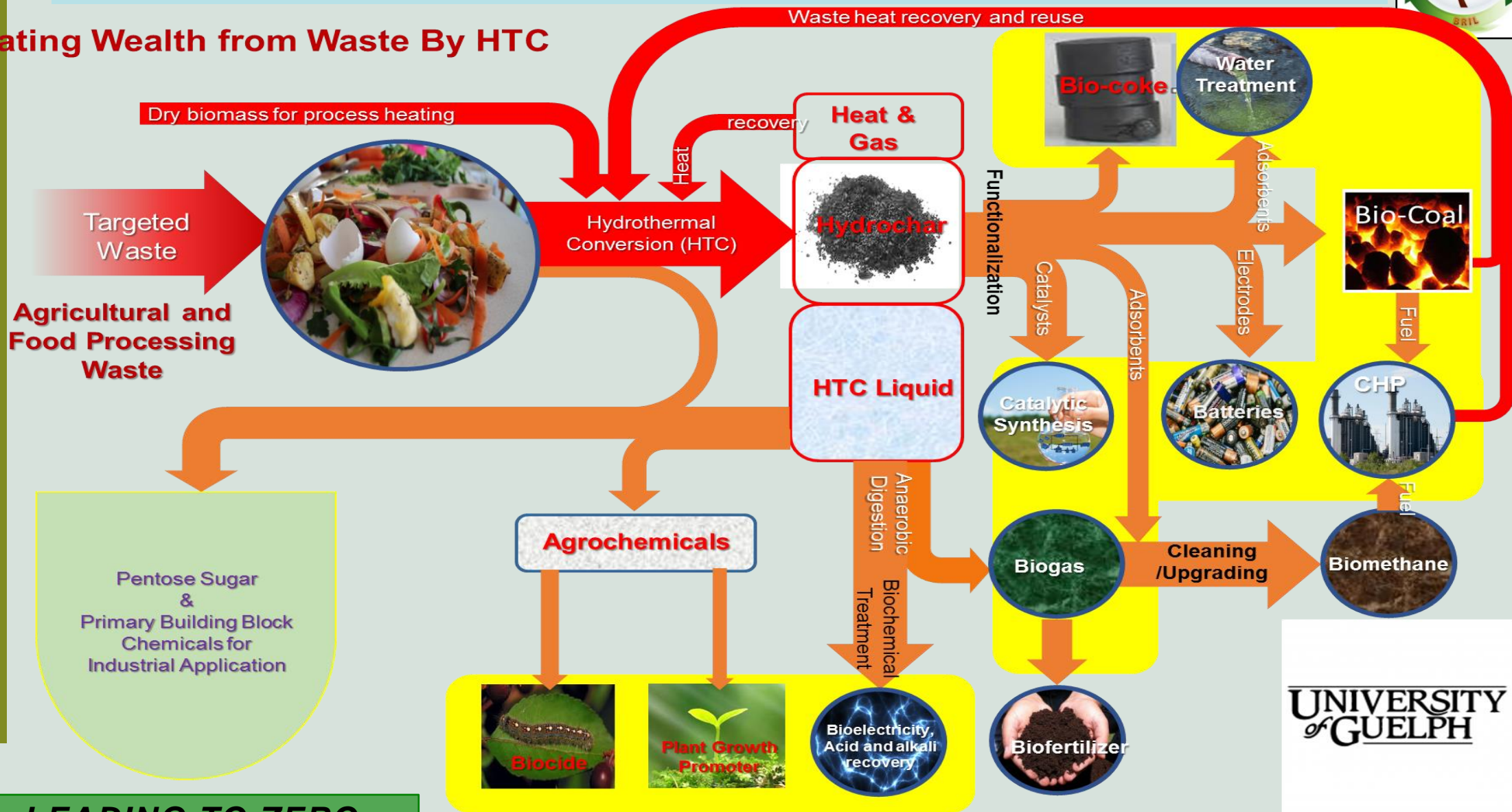
Research Project:

- Chemical Upcycling of Waste Plastic (USD OE) (CUWP.org) (Prof. George Hubber, University of Wisconsin-Madison, and Prof. Robert Brown, Iowa State University)
- Waste Plastic Pyrolysis: (Dr. Sultan al-Salem, KISR, Kuwait)
- Hydrothermal Carbonization research: (Dr. K Yoshikawa, Tokyo Institute of Technology, Japan, Dr. Kangil Chris Choe, Kinava, Korea, Dr. Brajesh Dube, IIT Kharagpur, India, Dr. Luca Fiori, Italy, Dr. Toufiq Reza, Florida Institute of Technology, USA)
- Renewable Methene (Dr. Francesco Di Maria, Universita di Perugia, Italy, Professor A. Nzihou of IMT-MA, France)

Approach: Preprocess greenhouse residues, energy crops, crop residues, municipal green bin fruit processing wastes through HTC processing.

Approach Under Evaluation

Creating Wealth from Waste By HTC



LEADING TO ZERO-WASTE SOLUTIONS

HTC products from low quality agri-food residue can be a potential newer value chain



BRIL TEAM



Acknowledgement



Thank You for Your Time
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