

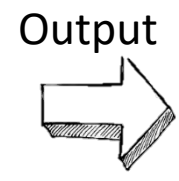
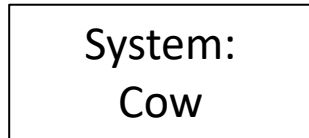
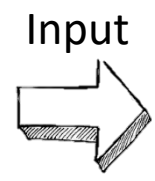
Valorisation of digestate through hydrothermal carbonization (HTC): a preliminary characterization of derived gaseous, liquid, and solid products

V. Benedetti¹, M. Pecchi¹, S. Celletti¹, D. Basso^{1,2}, F. Patuzzi¹, T. Mimmo¹, S. Cesco¹, M. Baratieri¹

¹ Faculty of Science and Technology, Free University of Bozen – Bolzano, Italy

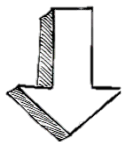
² HBI SrL, Italy







Biogas



- Electricity
- Heat

Anaerobic digestion plant



Digestate

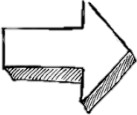


- Fertilizer

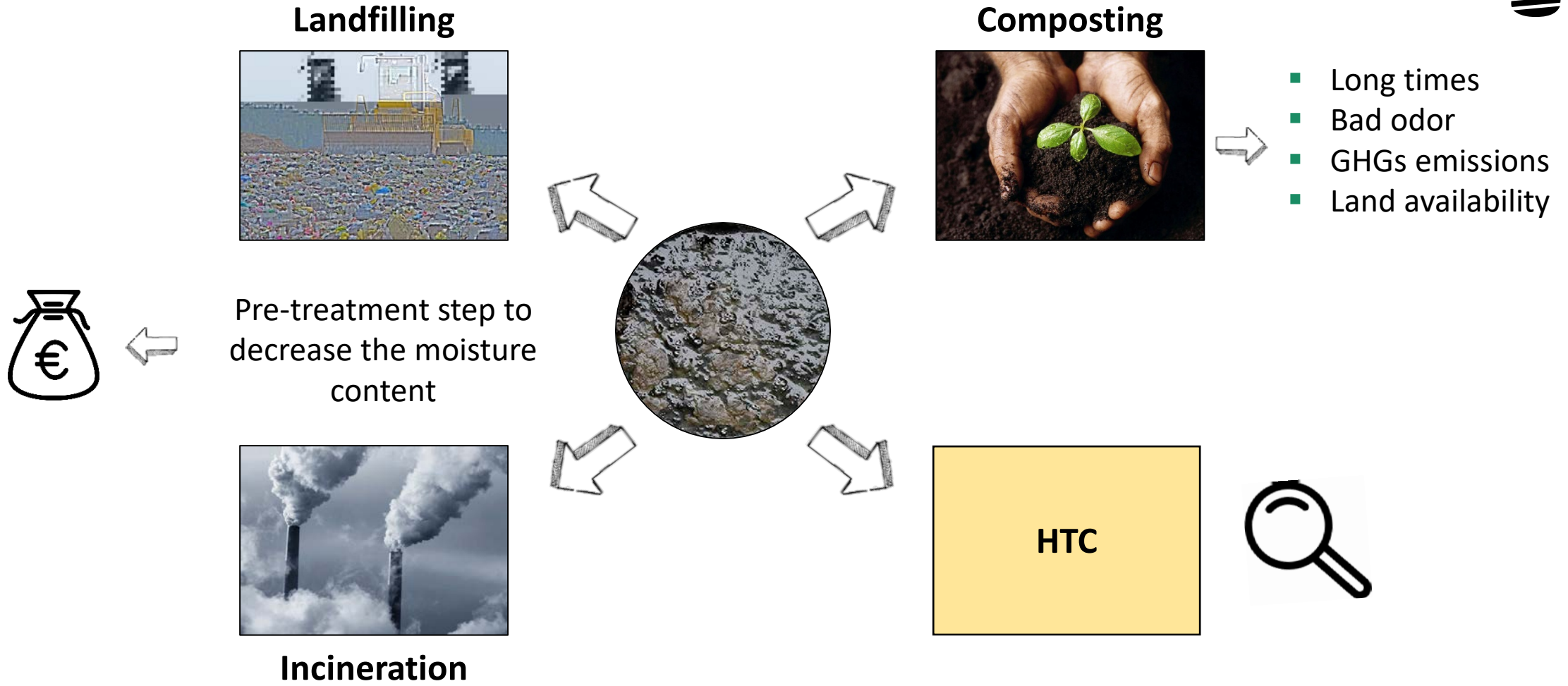




In Europe, Directives 2016/2284/EU and 91/676/EEC regulate the distribution of digestate on agricultural land, limiting the intake of N to $170 \text{ kg ha}^{-1} \text{ year}^{-1}$

- Water pollution: nitrate and nutrients leach into the groundwater causing eutrophication and hypoxia
- Air pollution: ammonia volatilization
- Very high water-content and residual biological activity  management issues
- Economic and environmental impacts







Hydrothermal carbonization (HTC)

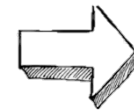
- Treatment of biomass in hot (180-250 °C) compressed water at residence times varying from minutes to several hours
- Ideal for biomass with high moisture content (> 60 %)
- Products:

- Hydrochar (HC)

- Aqueous HTC liquid (AHL)



Valorization





Hydrothermal carbonization



Hydrochar
(HC)



Bioenergy production

Soil amelioration

Carbon sequestration

Wastewater treatment

Carbon materials

Energy storage



Aqueous HTC Liquid
(AHL)



Anaerobic digestion

Fertilizers

Recirculation

Microalgae growth

P recovery



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Fondo europeo di sviluppo regionale



AUTONOME
PROVINZ
BOZEN
SÜDTIROL



PROVINCIA
AUTONOMA
DI BOLZANO
ALTO ADIGE



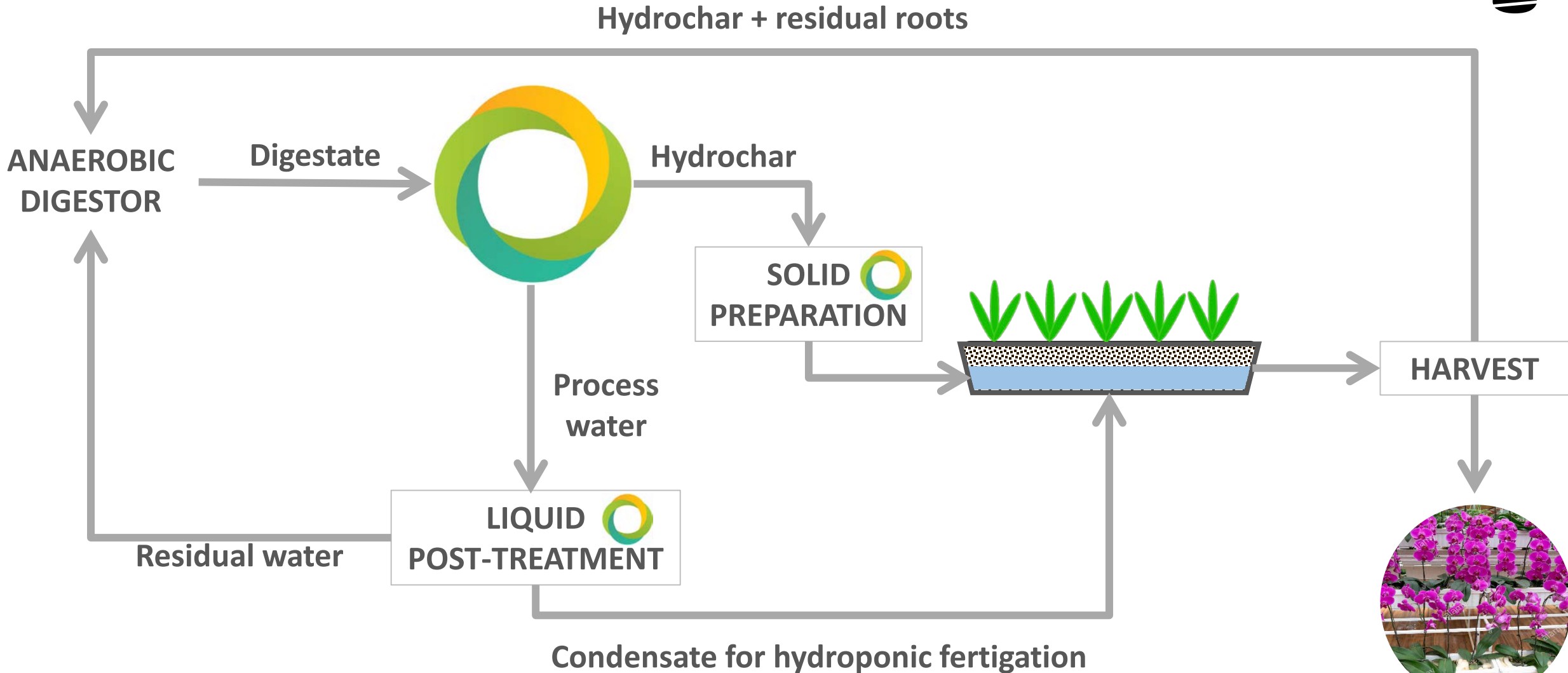
HB Ponics

Funding: Project **“HB Ponics”** (FESR1104; EFRE-FESR 2014-2020; CUP: B51B17000860008), financed by the European Regional Development Fund (ERDF) Investment for Growth and Jobs Programme 2014-2020.

<https://www.hbigroup.it/hb-ponics/>

Hydroponic cultivation

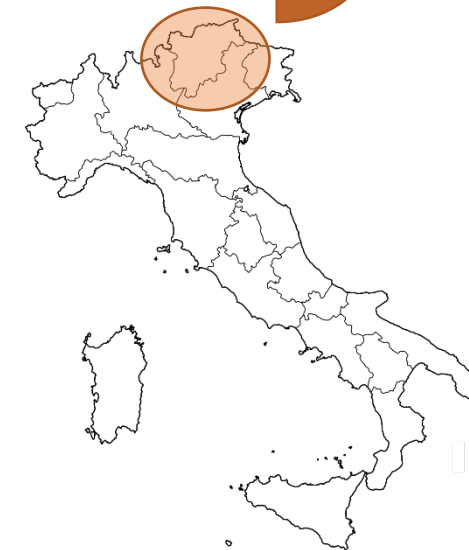
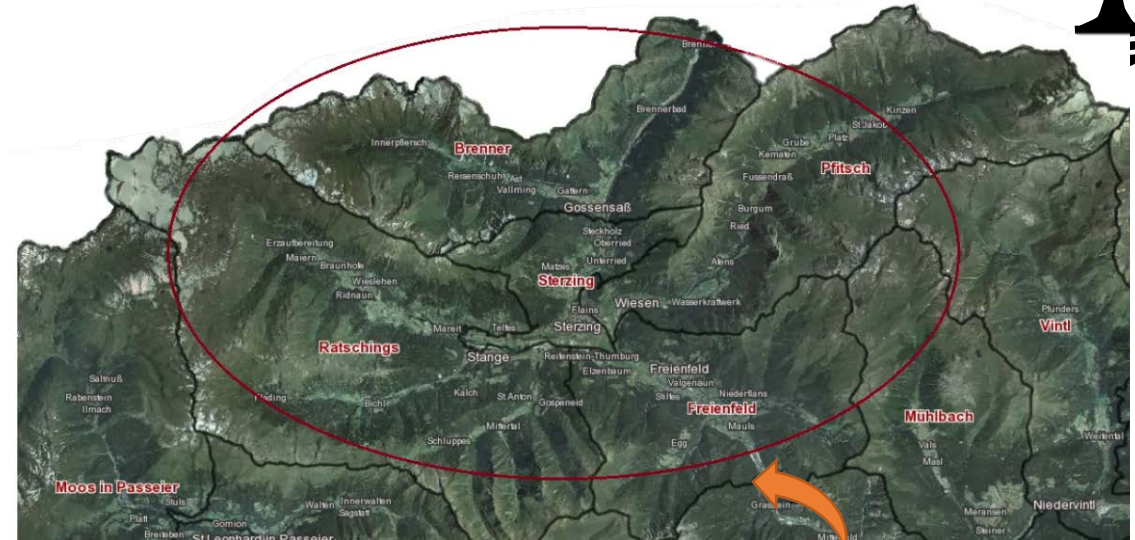




Digestate

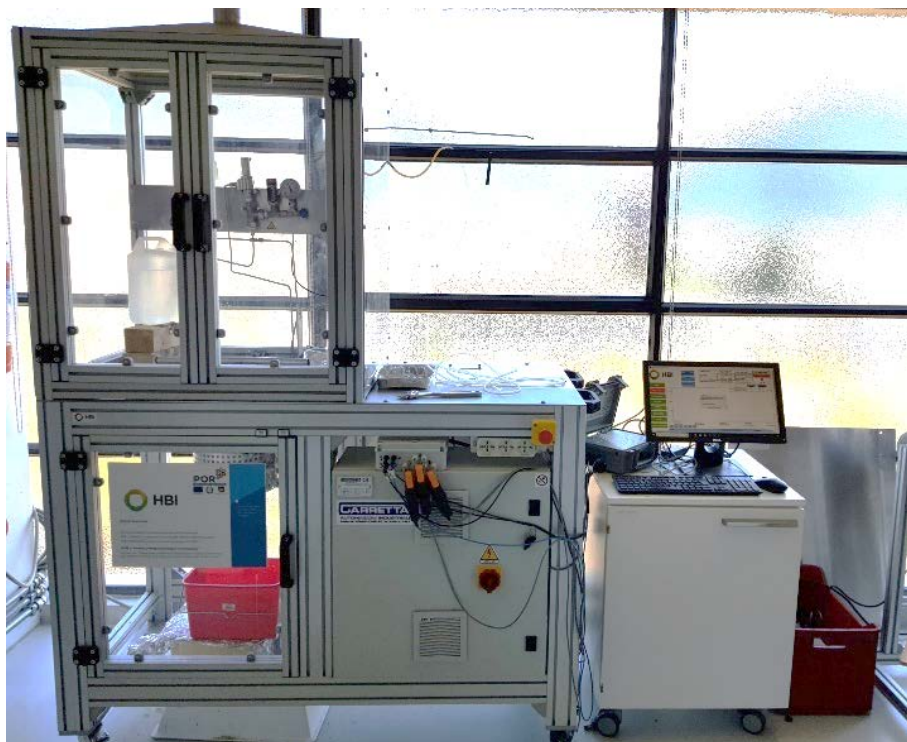


Source: courtesy of Biogas-Wipptal



Digestate		
Ash content	[%wt]	26.83
C	[%wt]	39.11
H	[%wt]	4.87
O	[%wt]	26.56
N	[%wt]	1.94
S	[%wt]	0.68
HHV	[MJ/kg]	14.31
LHV	[MJ/kg]	13.24

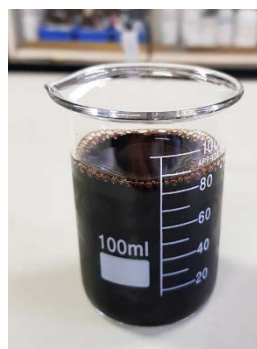
- 2.5 kg per experiment
- Previously kept in refrigerator at 4 °C
- No pre-treatment



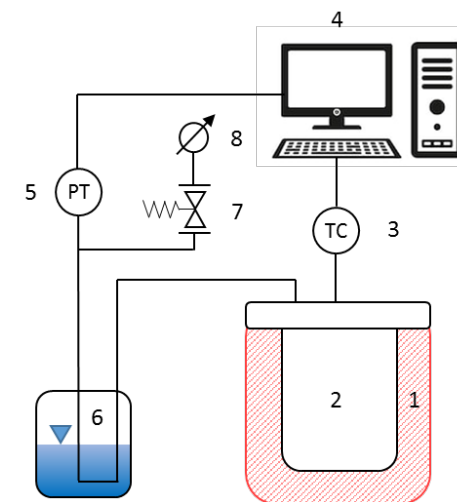
Batch reactor – 4 L



*Hydrochar – oven-dried
at 105 °C for 24 h*



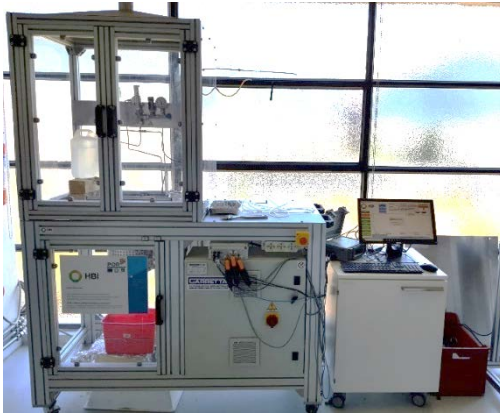
*Aqueous
HTC Liquid*



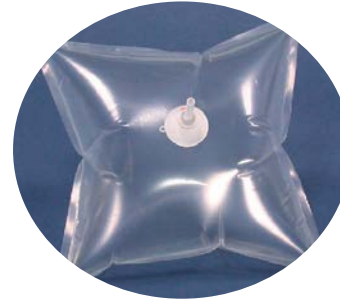
1. Electric furnace
2. HTC reactor
3. Temperature controller
4. HTC controller
5. Pressure transducer
6. Cold trap
7. Safety valve
8. Manometer

Scheme of the experimental lay-out

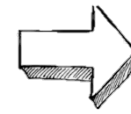
Operating condition	Experimental range		
Feedstock	digestate		
Temperature [°C]	180	220	250
Pressure	endogenous		
Residence time [h]	3		
Repetitions	3		



HTC reactor



Gas sampling bag



µGC

HTC temperature	CO ₂ %	H ₂ %	CH ₄ %	CO %
180 °C	65.49	0.04	0.26	0.12
220 °C	68.40	0.16	0.41	0.36
250 °C	79.18	0.35	0.70	0.30



TOC analysis



Feedstock	TOC
AHL 180 °C	[g/L] 7.07
AHL 220 °C	[g/L] 7.43
AHL 250 °C	[g/L] 7.89

Semi-continuous analysis

Spillages every 30 min during operation

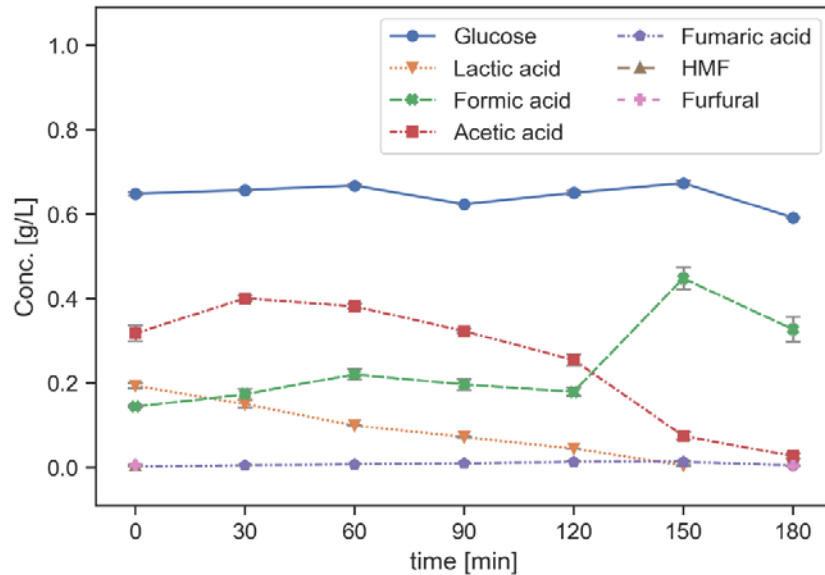


HPLC analysis

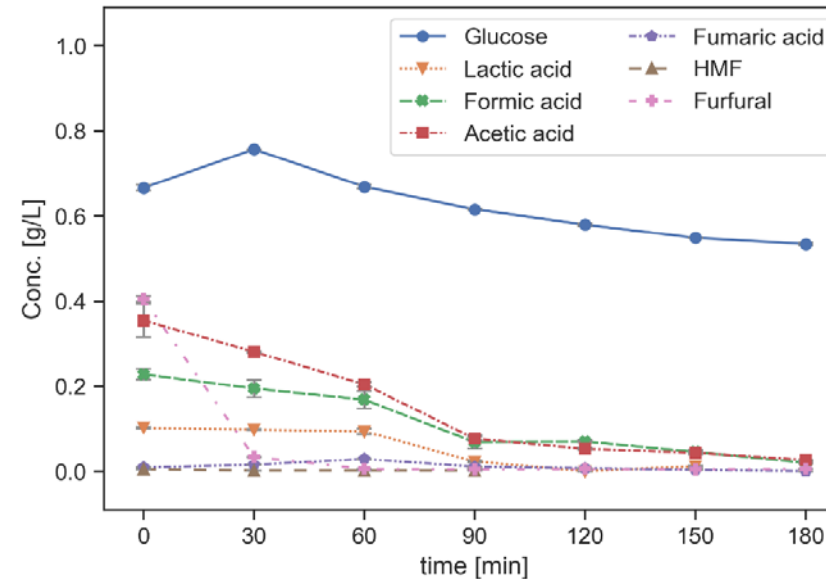
- Glucose
- Lactic, Formic, Acetic, Fumaric Acid
- Hydroxymethylfurfural (HMF), Furfural

AHLs characterization

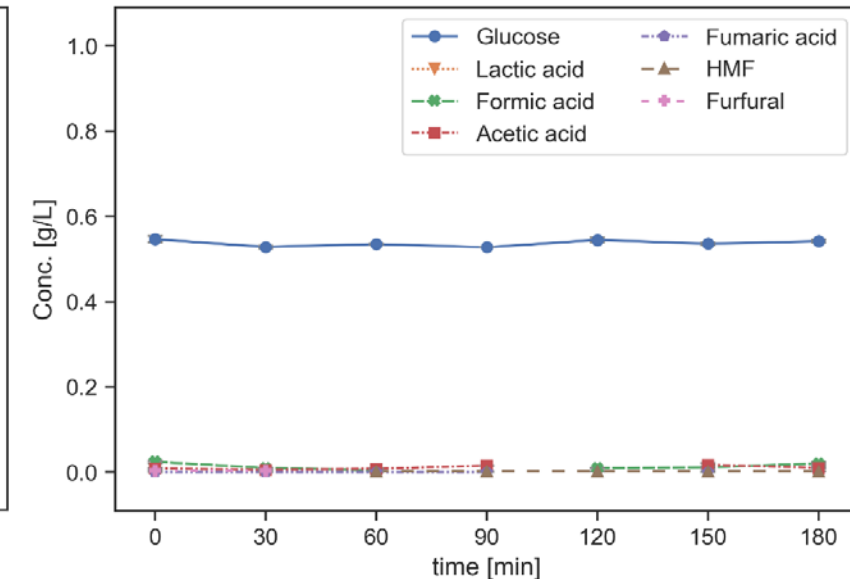
$T_{HTC} = 180\text{ }^{\circ}\text{C}$



$T_{HTC} = 220\text{ }^{\circ}\text{C}$



$T_{HTC} = 250\text{ }^{\circ}\text{C}$



- Increase of the reaction rate of **hydrolyzation** and **dehydration** that become the governing reactions of the process.
- Sugars, HMF and furfurals are **less stable at high temperature and residence time**.
- Polymerization and formation of secondary char.



Elemental analysis

1



Calorimetric analysis

2



Physisorption analysis

3



Germination tests

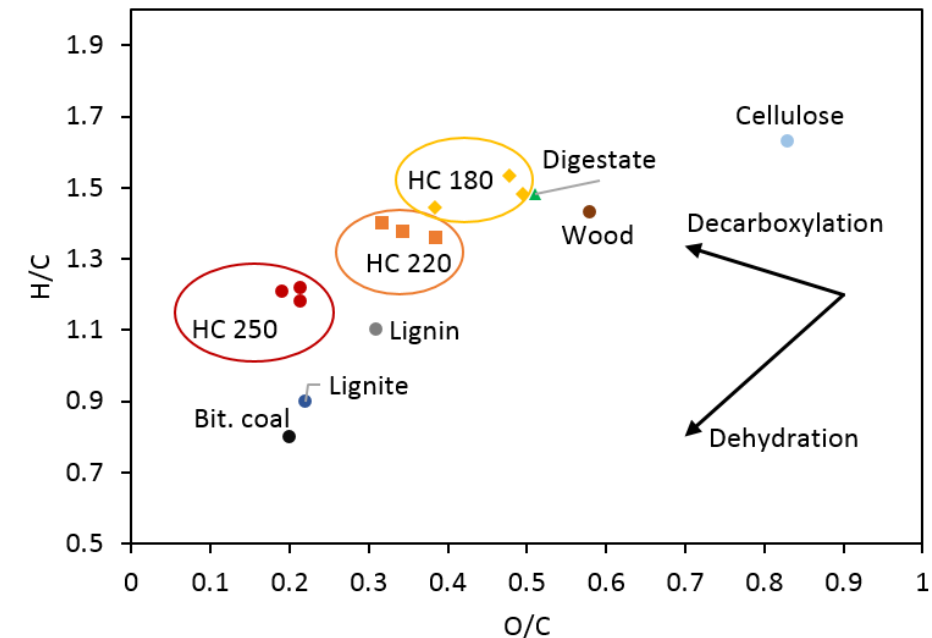
4

HCs characterization

	C	H	O	N	S	Ash	HHV
	%wt _{dry}	%wt _{dry}	%wt _{dry}	%wt _{dry}	%wt _{dry}	%wt _{dry}	MJ/kg
Digestate	39.11	4.87	26.56	1.94	0.68	26.83	14.31
HC 180	40.14	5.01	24.09	2.03	0.71	27.76	16.11
HC 220	42.53	4.92	19.71	2.16	0.62	29.79	16.70
HC 250	45.03	4.54	12.35	2.59	0.59	34.68	18.10

This is due to the **decarboxylation** and **dehydration** reactions that occur during the HTC process

Van Krevelen diagram





HCs characterization

	S_{BET}	Pore volume	Pore size
	m^2/g	cm^3/g	nm
Digestate	20	0.40	60
HC 180	35	0.79	56
HC 220	41	0.85	49
HC 250	52	0.86	47

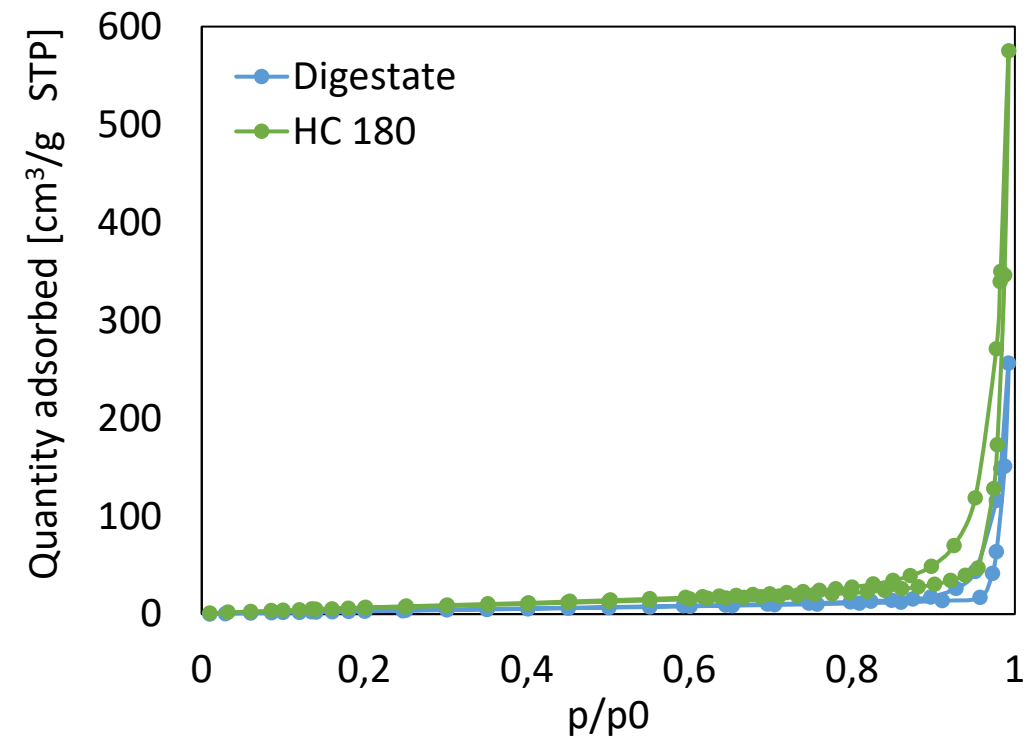
Type V adsorption-desorption isotherm

- Weak gas-solid interaction
- Characteristic of macro-porous materials

Type H3 hysteresis loop

- Indicative of slit pores

Adsorption-desorption isotherms (N_2 , 77 K)





HCs in soilless cultivation

Why?

Phytotoxicity tests on Cress (*Lepidium sativum* L.)

What?

- Hydrochars HC180, HC220, HC250.
- Water extracts at two different concentrations (5% and 10%, w/v) and two different pH (9 and 7).

How?

- Germination tests.
- Evaluation of plant morpho-physiological parameters (number of germinated seeds and total root length).
- Extracts characterization.

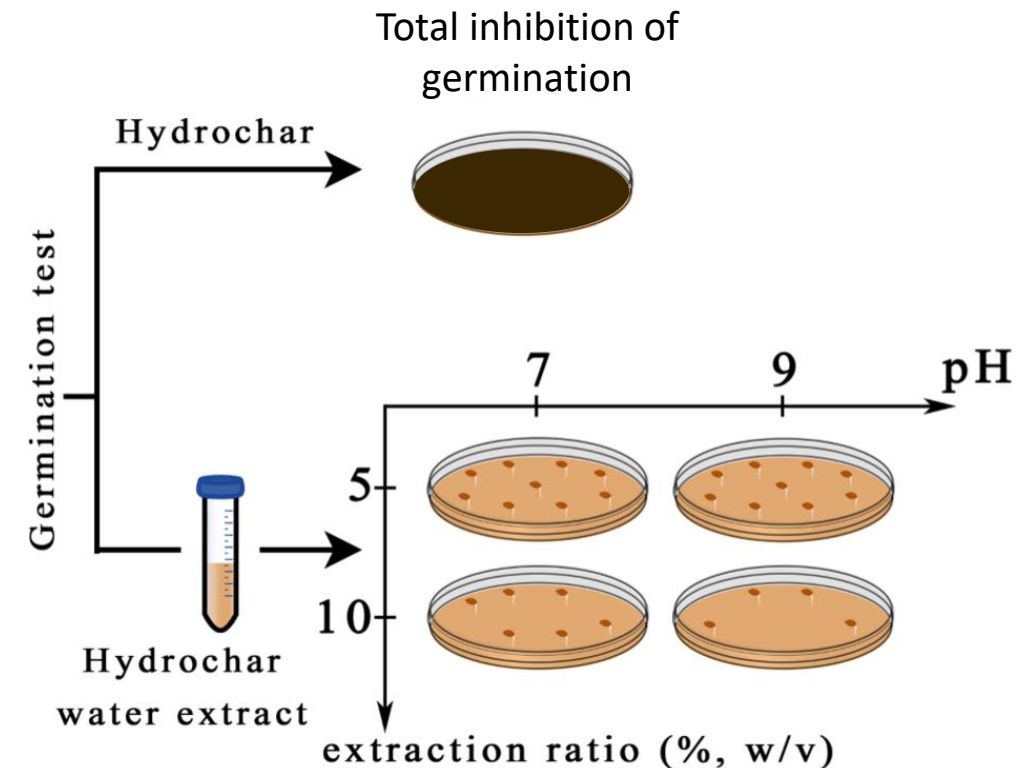


Cress (*Lepidium sativum* L.)

HCs in soilless cultivation

Main results

- Germination tests found hydrochar water extracts to show significantly lower phytotoxicity than the hydrochars themselves.
- The germination rate was reduced even further at higher process temperatures.
- The phytotoxic effect of the extracts decreased with decreasing extraction ratio and increasing pH.
- The chromatographic characterization of extracts identified the presence of potential phytotoxins, such as furan compounds.





HCs in soilless cultivation

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Research article

Phytotoxicity of hydrochars obtained by hydrothermal carbonization of manure-based digestate

Silvia Celletti ^{a,*}, Alex Bergamo ^a, Vittoria Benedetti ^a, Matteo Pecchi ^a, Francesco Patuzzi ^a,
Daniele Basso ^b, Marco Baratieri ^a, Stefano Cesco ^a, Tanja Mimmo ^a

^a Faculty of Science and Technology, Free University of Bolzano-Bozen, I-39100, Bolzano, Italy

^b HBI S.r.l., Bolzano, Italy





- HTC to treat and valorize digestate
- Temperature and residence time affect AHLs composition
- Temperature strongly affects HC characteristics
- HCs in soilless culture systems (SCS)
- The phytotoxic effect of the extracts decreased with decreasing extraction ratio and decreasing pH
- To reduce phytotoxicity, it could be advantageous to mix hydrochars with other types of growing media and verify their suitability by conducting growth experiments with plant species usually cultivated in SCS

Thank you for your attention

Valorisation of digestate through hydrothermal carbonization (HTC): a preliminary characterization of derived gaseous, liquid, and solid products

E-mail: vittoria.benedetti@unibz.it

Website: <https://bnb.groups.unibz.it/>

