

# THESSALONIKI2021

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**Influence of cavitation, pelleting, extrusion and torrefaction pretreatments on anaerobic biodegradability of barley straw and vine shoots**

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# The BIOMETRANS project

“Production of biomethane for transport fuel from biomass waste”



The BIOMETRANS project aims to promote the valorisation of dry and wet biomass residues that are generated in the Ibero-American Region, through the production of biomethane and promote its use as a transport biofuel.



Out of all bioconversion technologies for production of biofuels, **anaerobic digestion** is one of the most profitable and this is the reason why it has been implemented worldwide for the commercial production of electricity, heat and compressed natural gas (CNG) from all kind of organic materials, including waste.



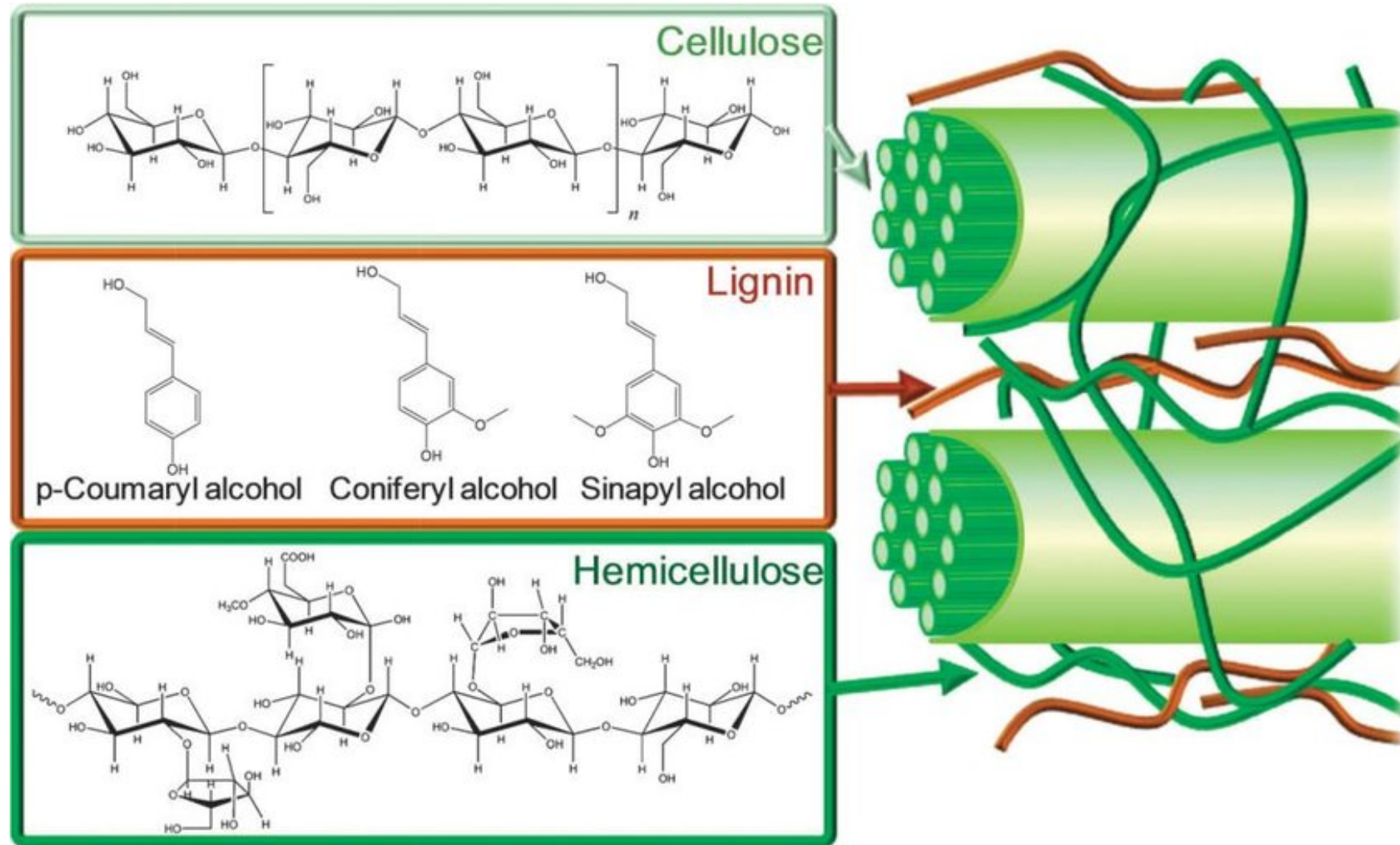
On the other hand, large quantities of **lignocellulosic biomass** that accumulate from agricultural, forestry and other activities are available for the sustainable production of biofuels.



However, the use of **lignocellulosic biomass for the production of biogas** through anaerobic digestion has not been widely adopted because the inherent structure and chemical properties of the plant cell wall makes it resistant to microbial attack.

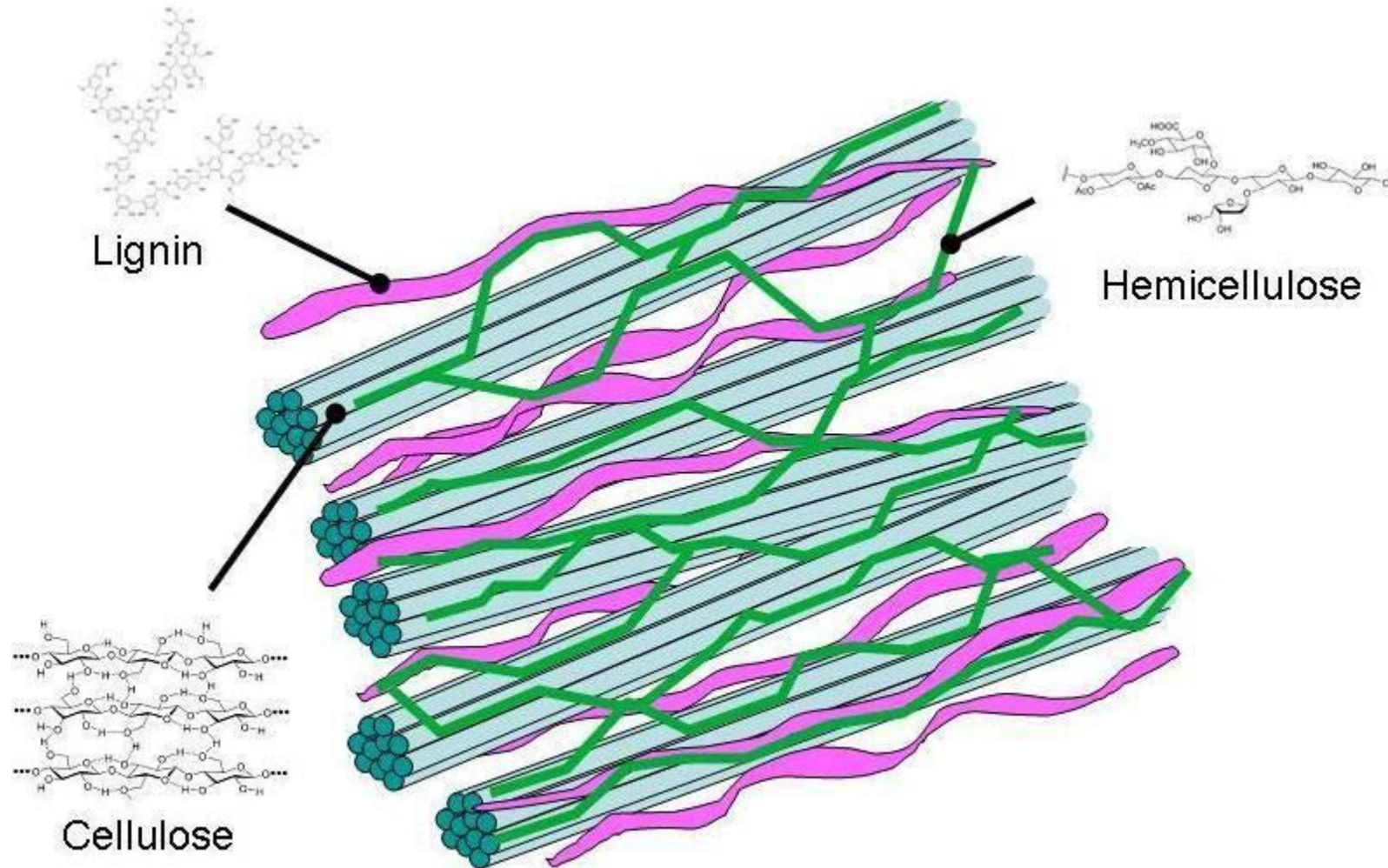


# Rationale



Lignocellulosic biomass is mainly composed of three components: cellulose, hemicellulose and lignin.

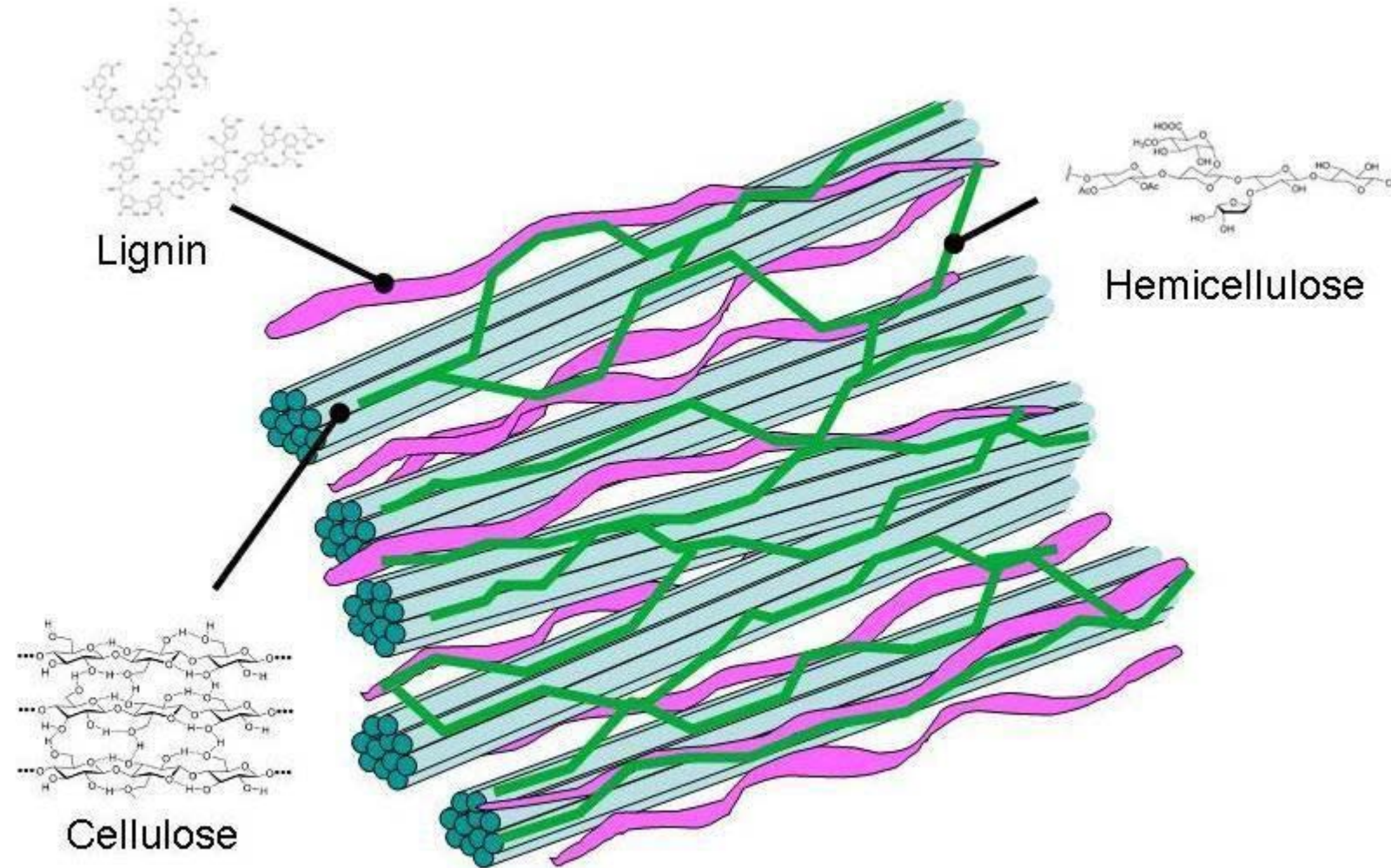
# Rationale



Cellulose and hemicellulose are the main carbohydrate components of the biomass structure.



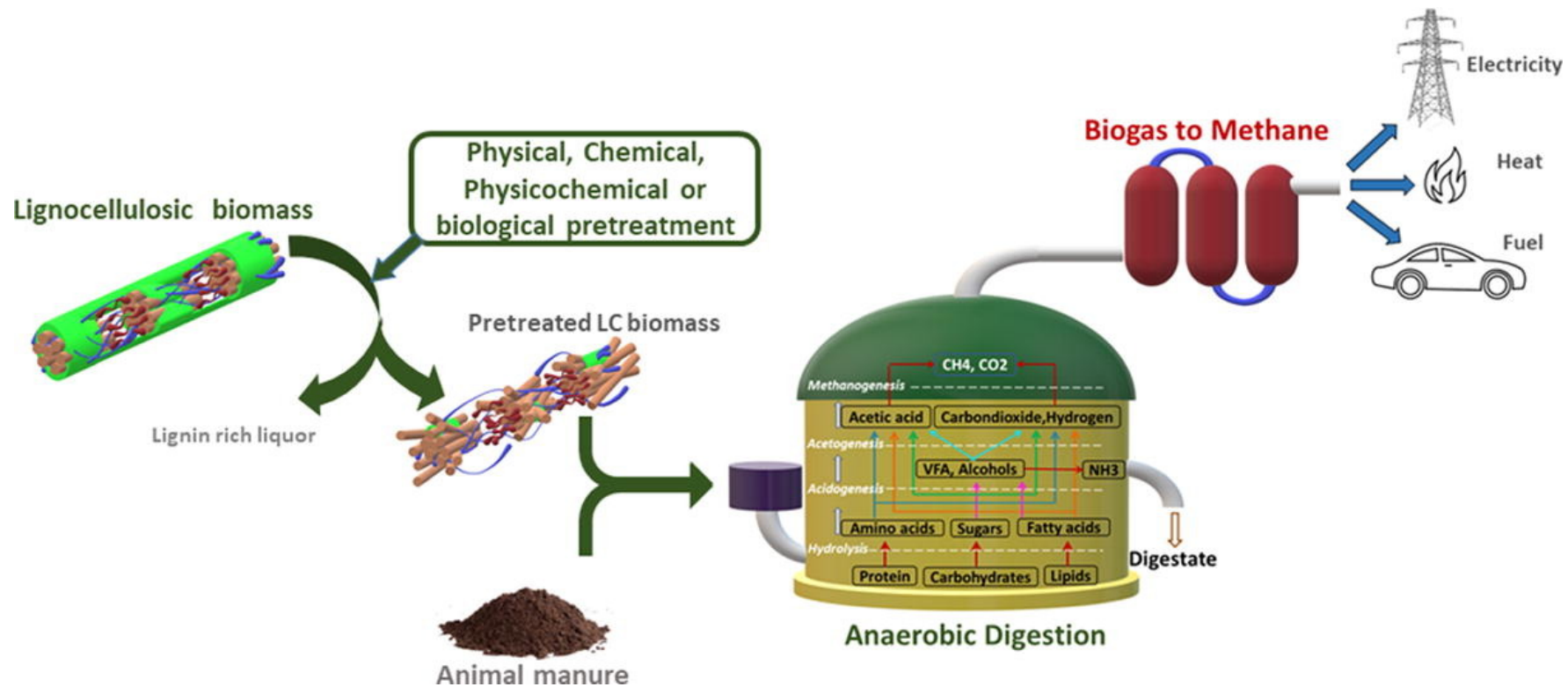
# Rationale



Lignin is a highly cross-linked polyphenolic macromolecule that provides rigidity to the cell wall of the material.

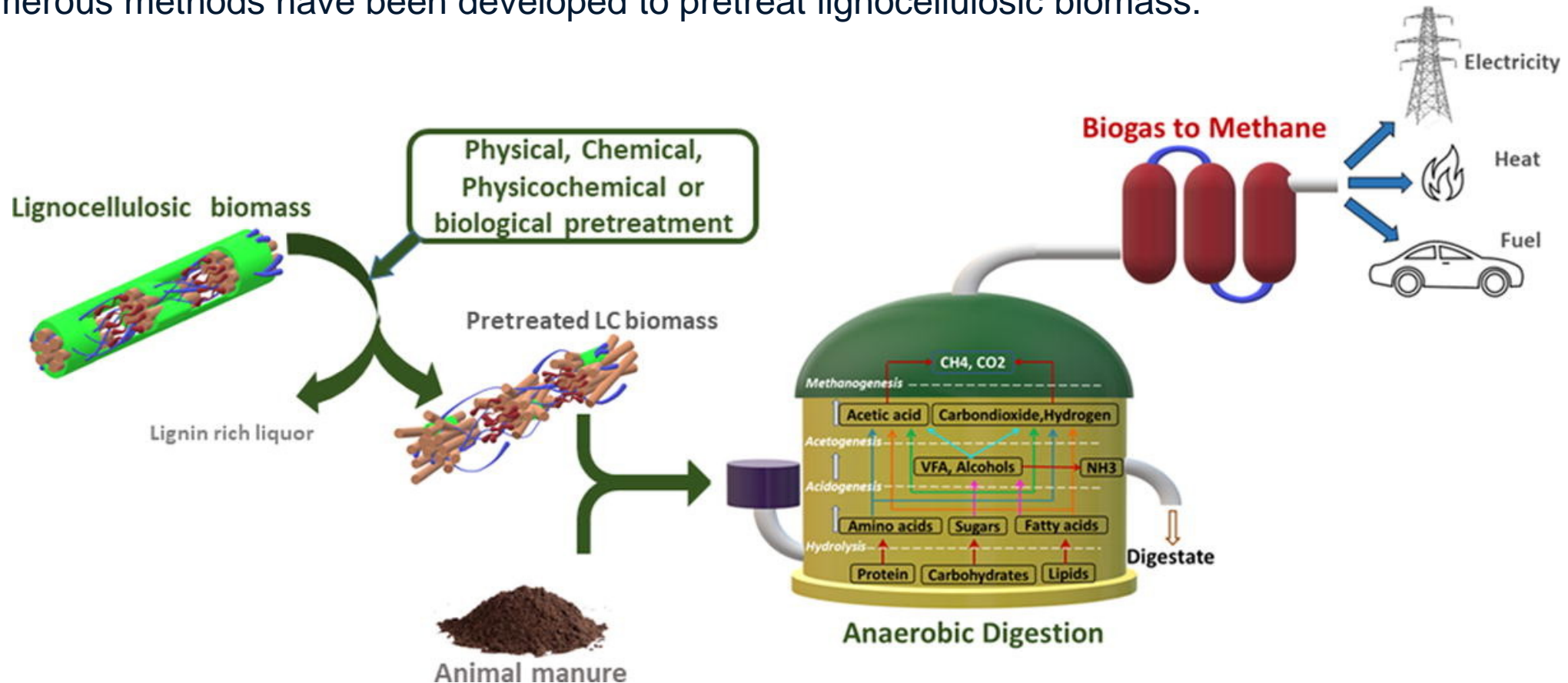
# Rationale

- To recover the monomeric sugars present in the carbohydrate fractions for later use in bioprocesses, a **pretreatment stage** is required to open the structure of the material, facilitating its degradation by enzymes and microbes.
- Numerous methods have been developed to pretreat lignocellulosic biomass:



# Rationale

- To recover the monomeric sugars present in the carbohydrate fractions for later use in bioprocesses, a **pretreatment stage** is required to open the structure of the material, facilitating its degradation by enzymes and microbes.
- Numerous methods have been developed to pretreat lignocellulosic biomass:





# Proposed work

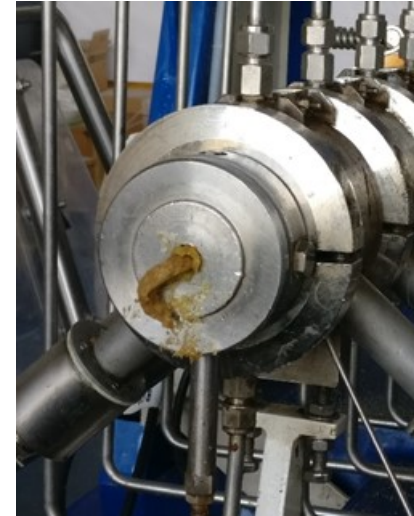
Four methods to pretreat lignocellulosic biomass have been evaluated:



Cavitation



Pelleting



Extrusion



Torrefaction

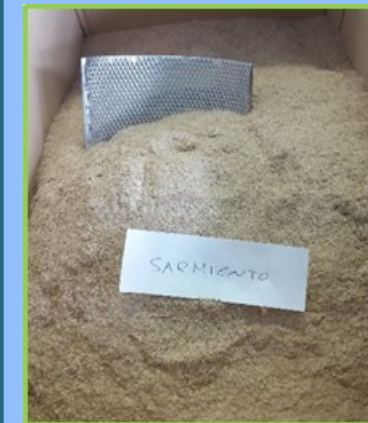
With the aim to improve the production of methane by anaerobic digestion of:

- Barley straw
- Vine shoot



# Milling (first step)

Prior to any pretreatment, the biomass was **mechanically milled** so that they reached an adequate particle size. The equipment used was a blade mill for the straw and a hammer mill for the vine shoot.



# Cavitation

**Hydrodynamic cavitation (HC)** is an undesirable phenomenon for hydraulic equipment, but the net **energy released during this process is sufficient to accelerate certain chemical reactions.**

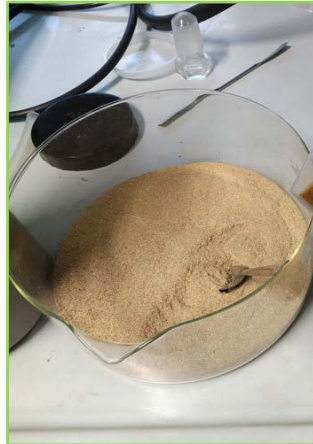
The application of cavitation energy to improve the efficiency of pretreatment of lignocellulosic biomass is an interesting strategy.



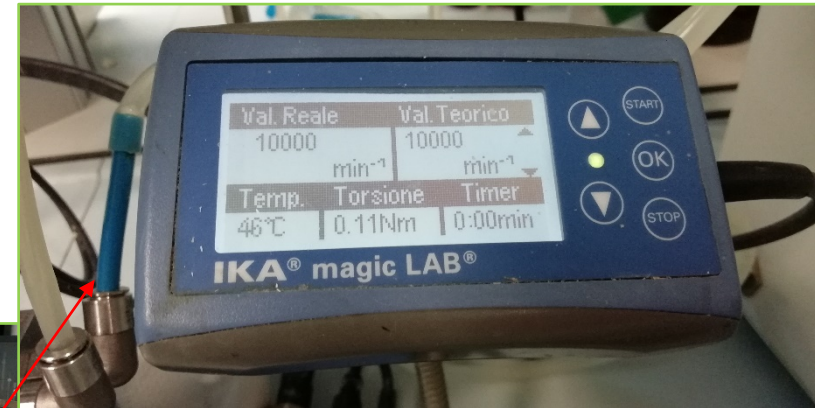
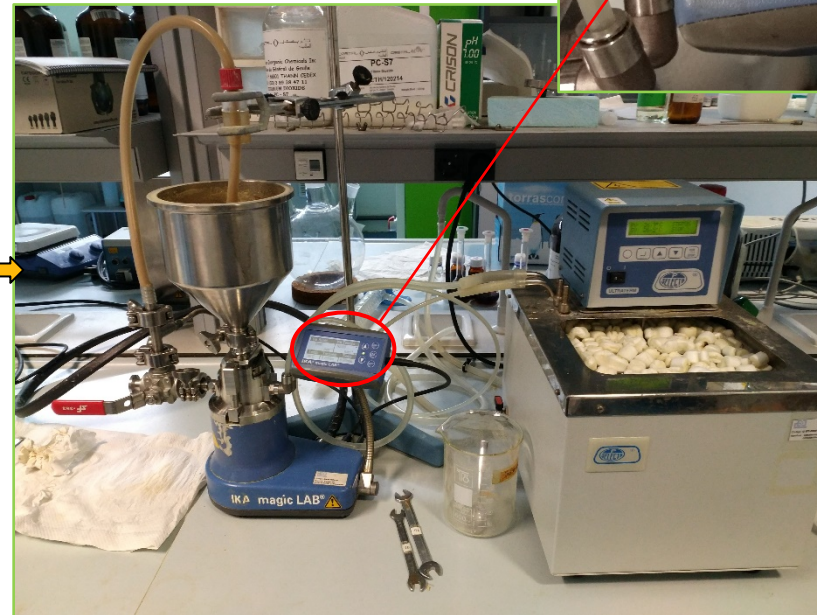
The beneficial effects of HC are attributed to the **release of large amounts of energy in a very small location** ("hot spots") during the collapse of the cavities, resulting in very high energy densities due to the generation of local conditions of very high temperature and pressure.



# Cavitation



$D = 0,25 \text{ mm}$   
 $T^a \text{ max} = 50 \text{ }^\circ\text{C}$



# Densification

Biomass densification, as **pelleting** or **extrusion**, can be employed to reduce biomass volume and facilitate logistic in biomass management activities.

**Pelleting** of biomass involves **size reduction and conditioning of the ground biomass** by applying heat and/or pressure. These operations may hydrolyze the hemicellulose and lignin into lower molecular carbohydrates, sugar polymers and other derivatives.



$D = 2 \text{ mm}$   
 $T^a \text{ max} = 90 \text{ }^\circ\text{C}$

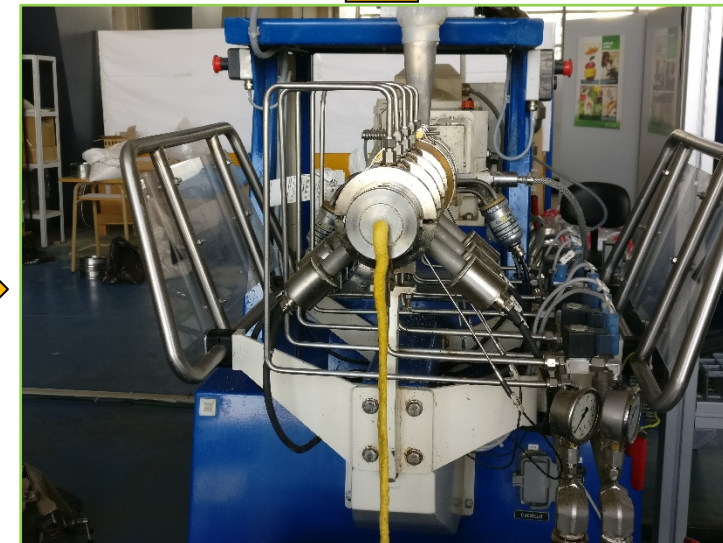


# Densification

**Extrusion** process submits biomass to heat by friction, mixing and vigorous shear by releasing the pressure at the end. Extrusion can significantly **reduce the particle size, increasing the specific surface area** of the biomass and causing depolymerization of cellulose.



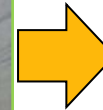
$D = 2 \text{ mm}$   
 $T^a \text{ max} = 100 \text{ }^\circ\text{C}$





# Torrefaction

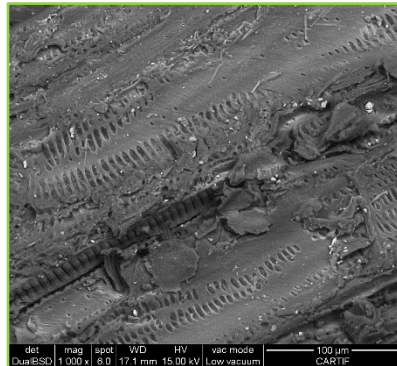
**Torrefaction** is a thermochemical pretreatment method taking place at temperatures ranging up to 300 °C. At these temperatures the skeleton of lignin and hemicellulose can be broken down partly, which is an interesting effect that can favour AD process.



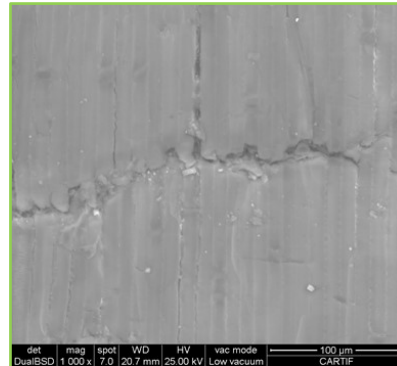
D = 2 mm  
T<sup>a</sup> 1 = 220 °C  
T<sup>a</sup> 2 = 120 °C

# SEM Analysis

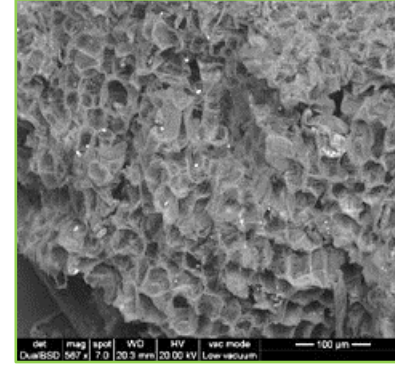
The SEM images of the untreated biomass show the robust structure and relatively smooth surface in straw and vine shoot fibers.



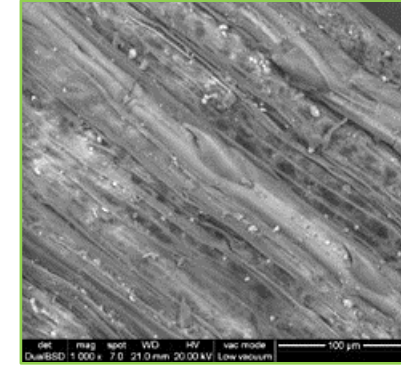
• Vine shoot no-T



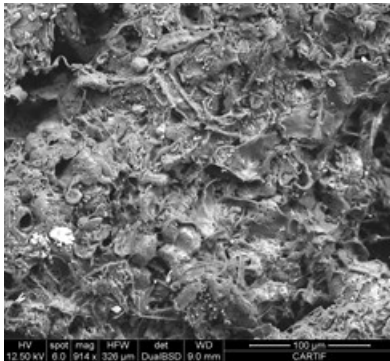
Straw no-T



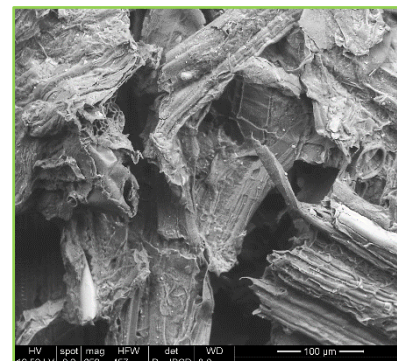
• Vine shoot Torr.



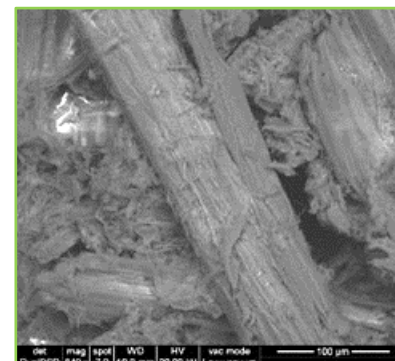
Straw Torr.



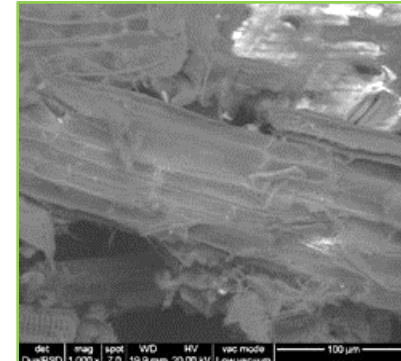
• Vine shoot Ext.



Straw Ext.



• Vine shoot Cav.



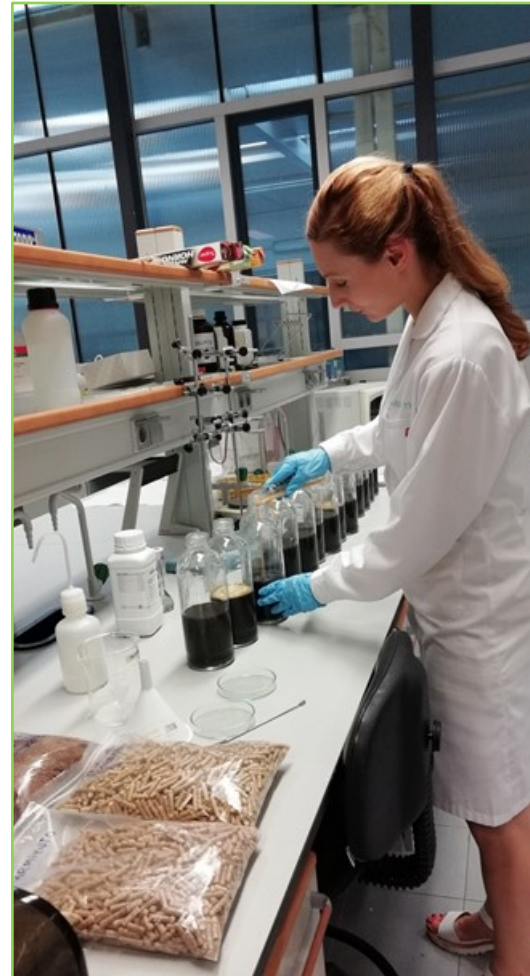
Straw Cav.

The ability of the different pretreatments to create **structural damages** on the biomass morphology is observed. The structure became loose and disordered.



# BMT Analysis

In order to study the effect of the different pretreatments on the biodegradability of the biomass, batch experiments were run in glass serum bottles with a liquid volume of 500 mL.





# BMT Analysis

All the experiments were carried out at  $34\pm 1$  °C in a thermostatic room.

Digestate from an anaerobic reactor operating in a municipal wastewater treatment plant, with a VS concentration of  $7,1\pm 0,5$  g/L, was used as inoculum for the anaerobic test.

Final inoculum concentration in the tests medium was  $5.0\pm 0.5$  g/L.

The substrate/inoculum (S/X) ratio was maintained for all samples at  $0.50\pm 0.05$  gSV<sub>substrate</sub> / gSV<sub>inoculum</sub>.

Biogas production was automatically measured by using pressure transmitters.

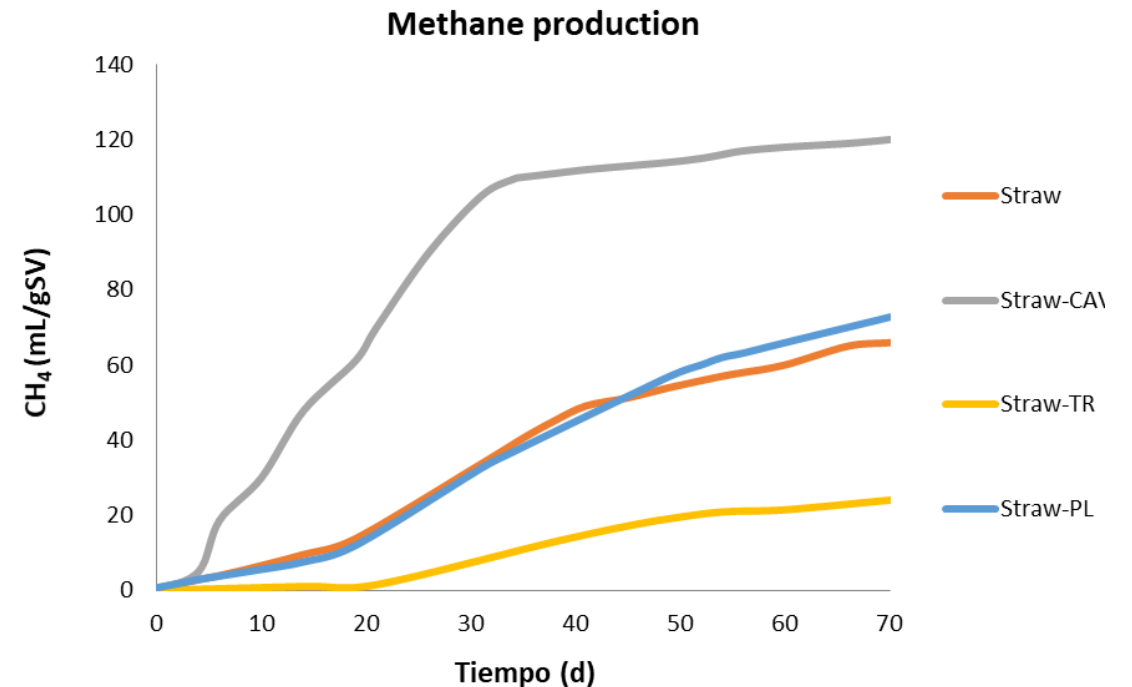
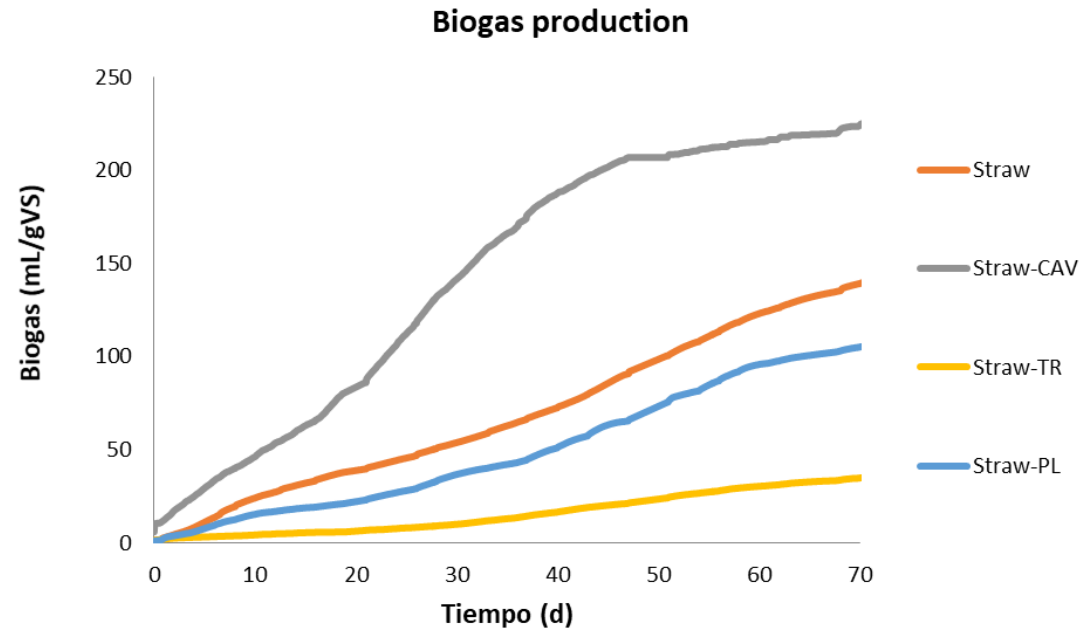
The biogas composition was measured using a Micro-GC chromatograph with a Thermal Conductivity Detector.

The individual Volatile Fatty Acids (i.e., acetic, propionic, isobutyric, butyric, isovaleric and valeric acids) were quantified using GC equipped with flame ionization detector (GC-FID).

# First results

**Exp. 1:** Digestion of milled cereal straw using WWTP digestate as inoculum

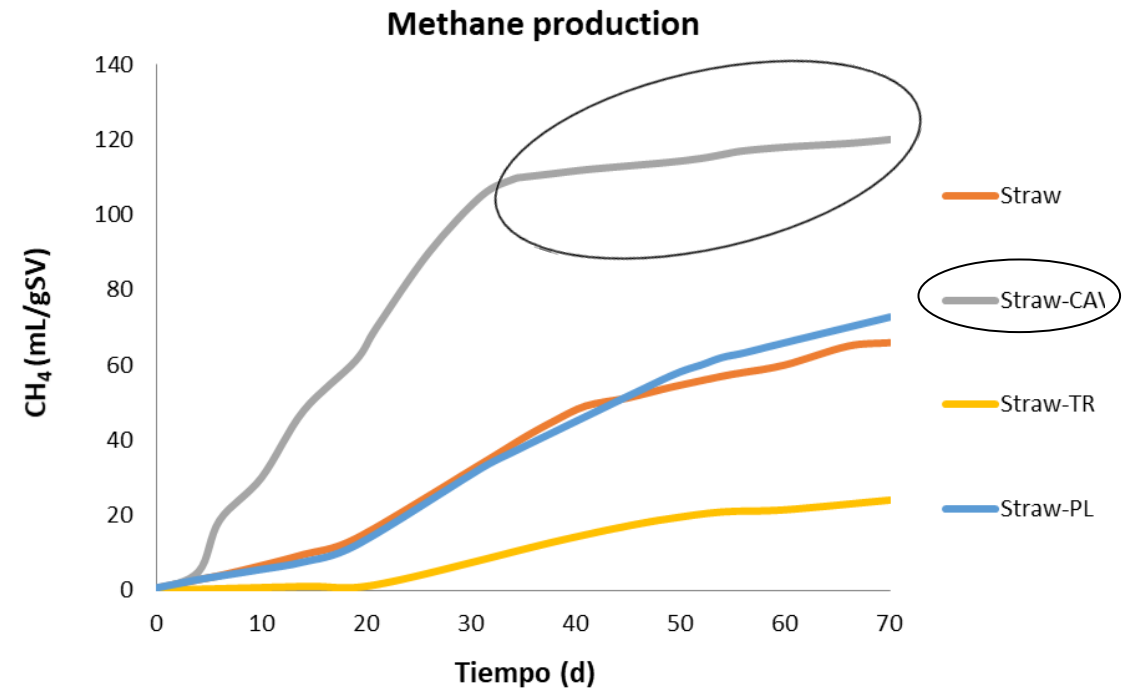
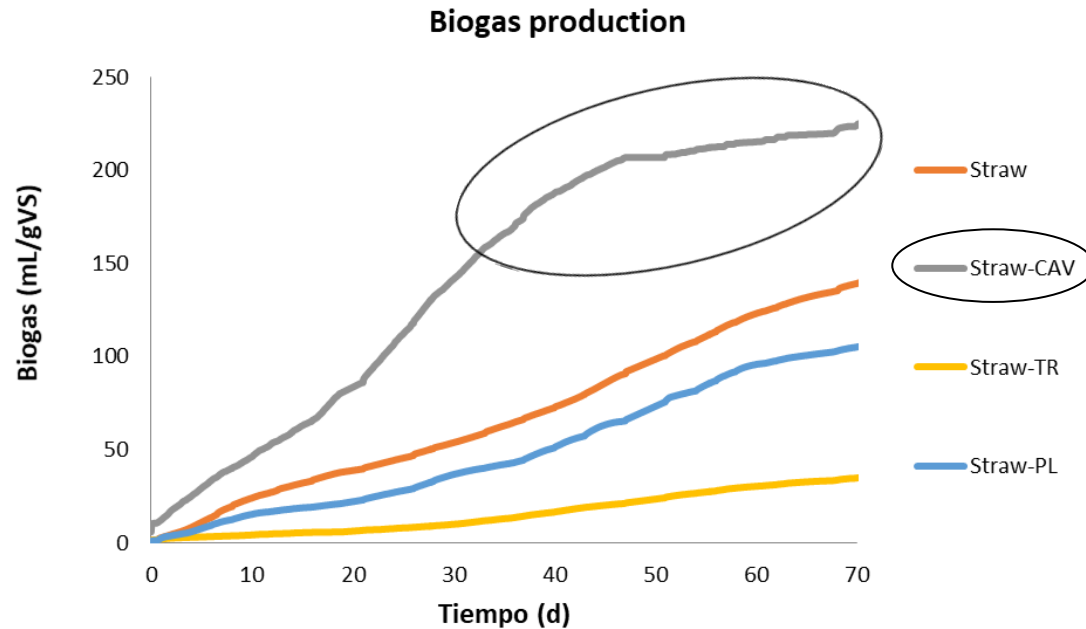
Pretreatments: Cavitation (CAV), Torrefaction at 220 °C (TR) and Pelleting (PL)



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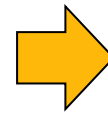
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T = 0

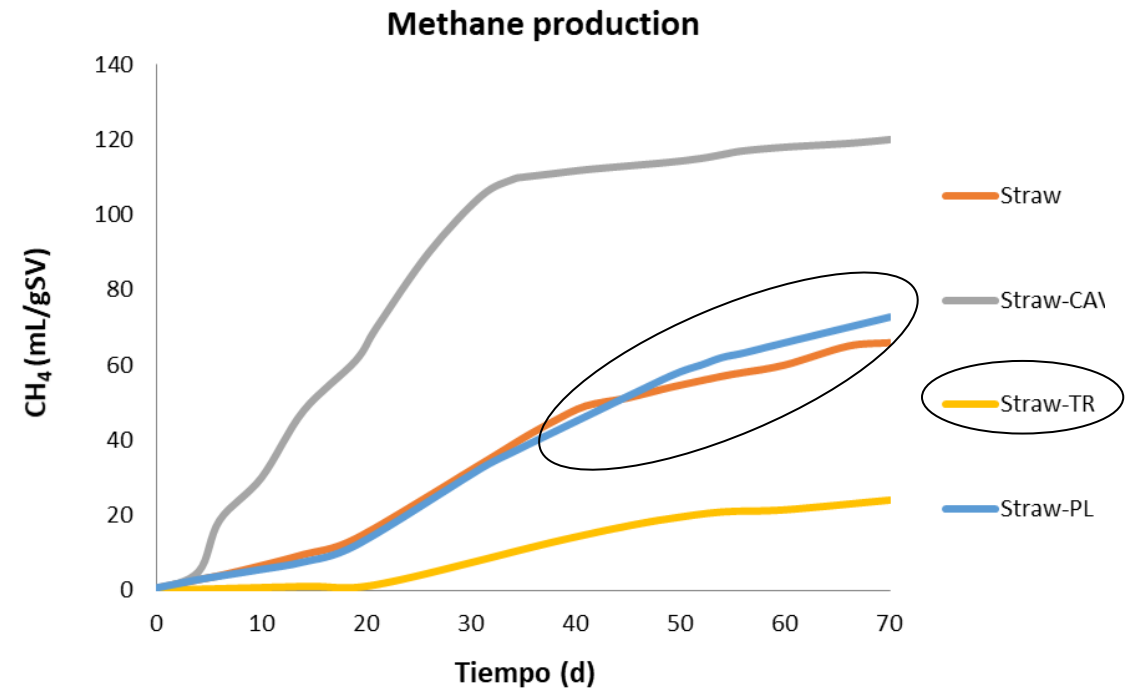
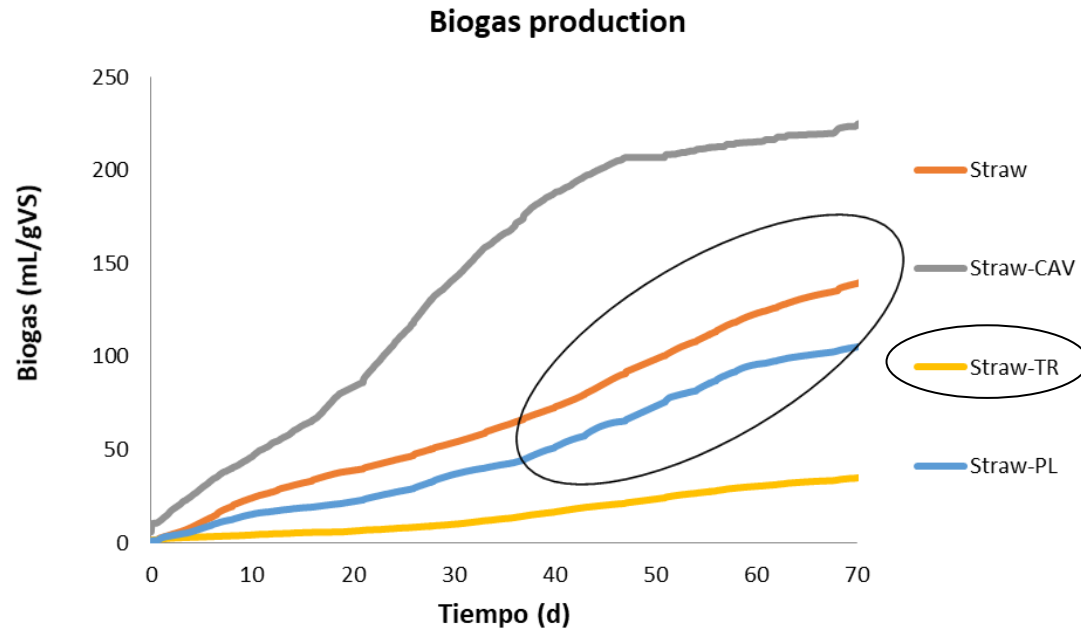


T = 3 days mold colonies

# First results

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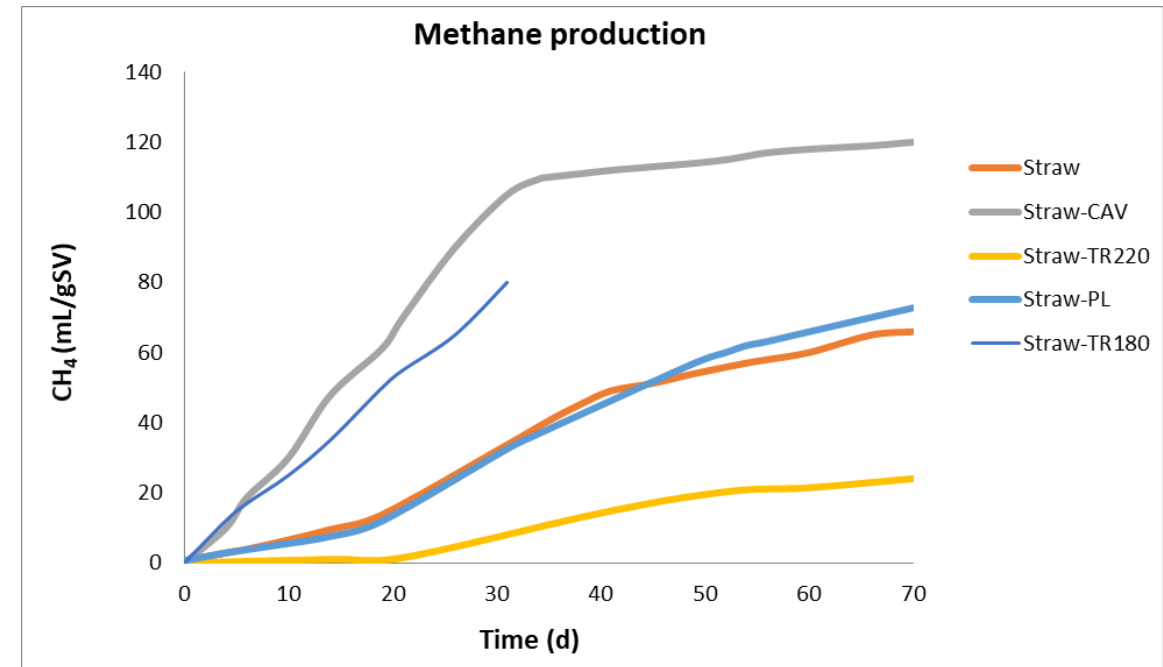
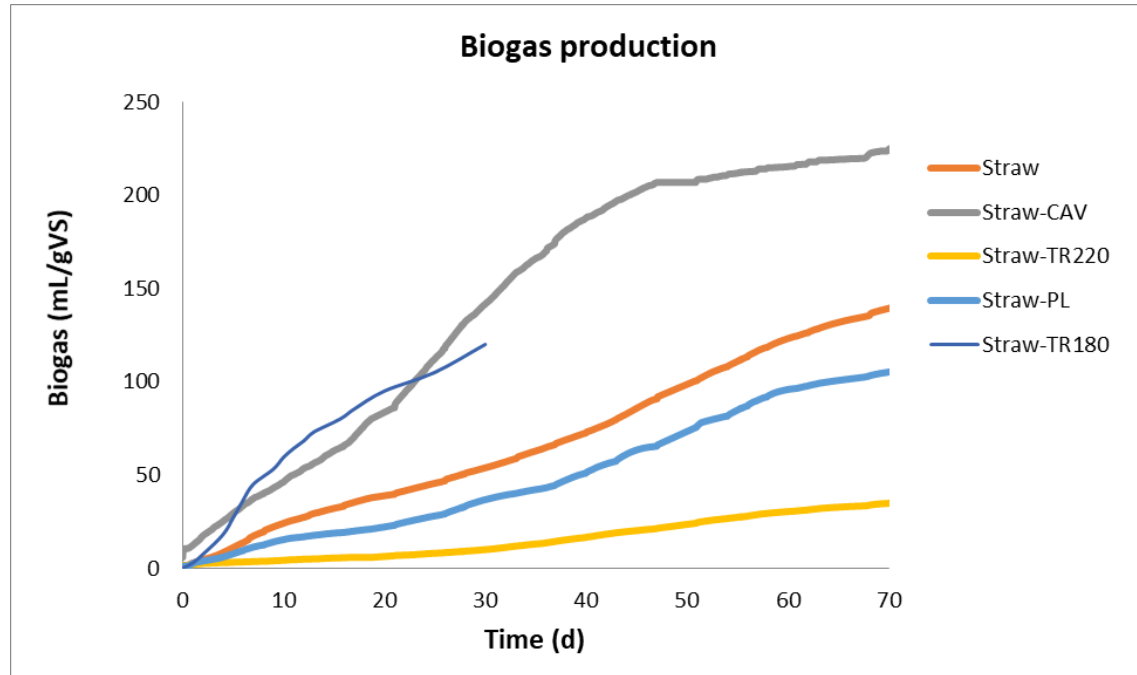
Pretreatments: Cavitation (CAV), Torrefaction at 220 °C (TR) and Pelleting (PL)



# First results

**Exp. 2:** Digestion of milled cereal straw using WWTP digestate as inoculum

Pretreatments: Torrefaction at 180°C (TR180) vs Torrefaction at 220°C (TR220)

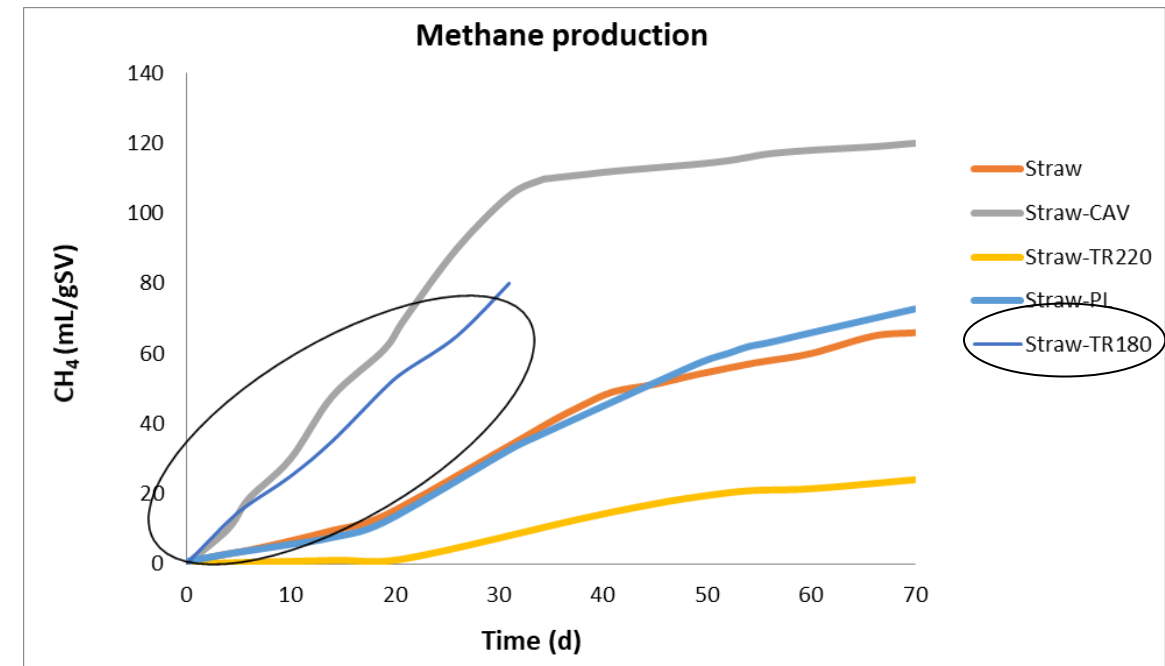
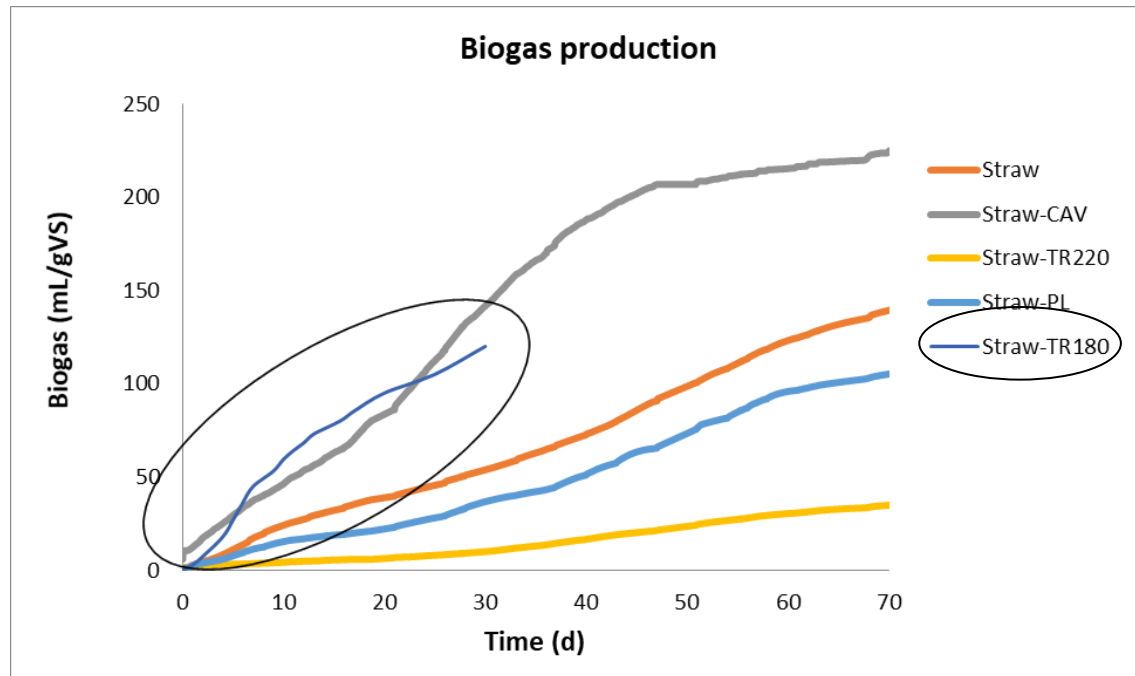




# First results

**Exp. 2:** Digestion of milled cereal straw using WWTP digestate as inoculum

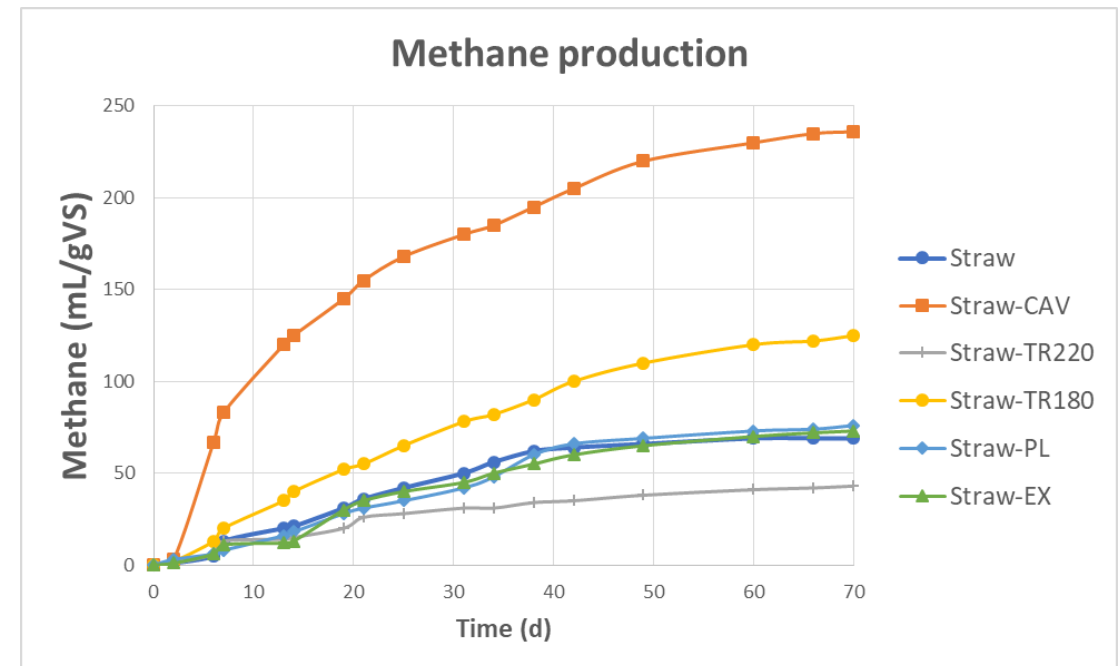
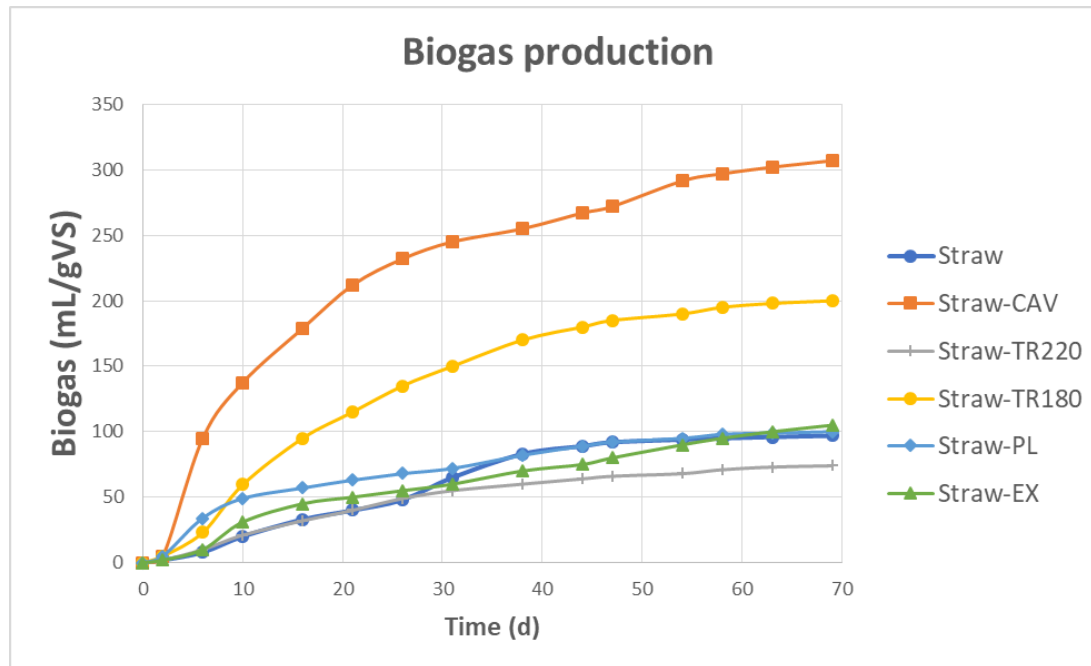
Pretreatments: Torrefaction at 180°C (TR180) vs Torrefaction at 220°C (TR220)



# First results

**Exp. 3:** Digestion of milled cereal straw using WWTP digestate (fresh) as inoculum

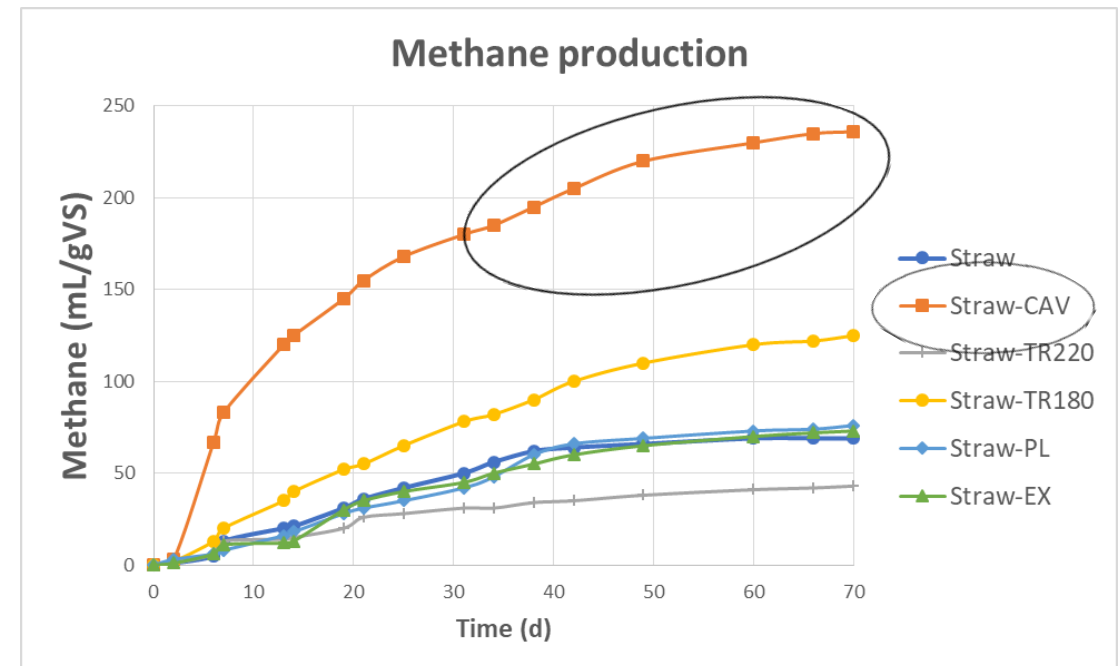
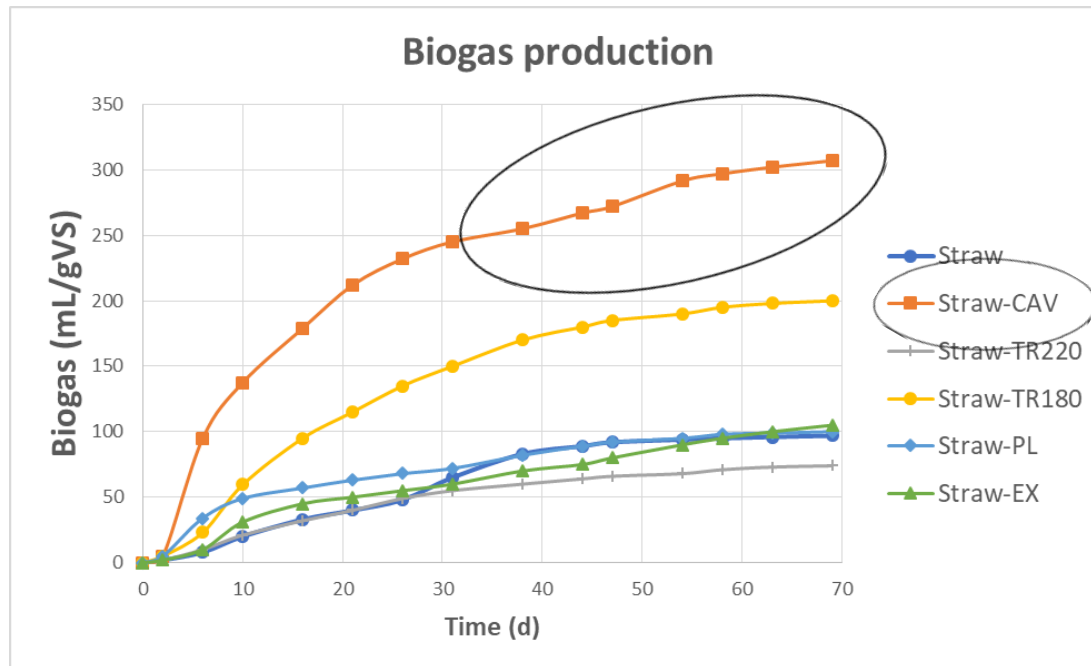
Pretreatments: Cavitation (CAV), Torrefaction at 220°C (TR220), Torrefaction at 180°C (TR180), Pelleting (PL) and Extrusion (EX)



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**Exp. 3:** Digestion of milled cereal straw using WWTP digestate (fresh) as inoculum

Pretreatments: Cavitation (CAV), Torrefaction at 220°C (TR220), Torrefaction at 180°C (TR180), Pelleting (PL) and Extrusion (EX)

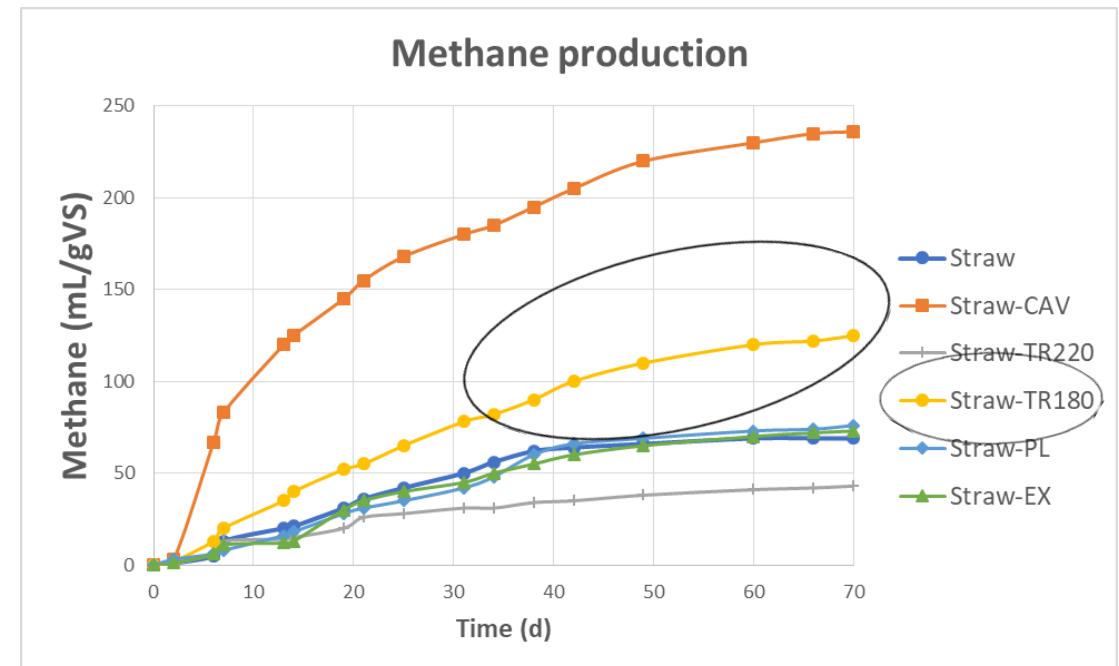
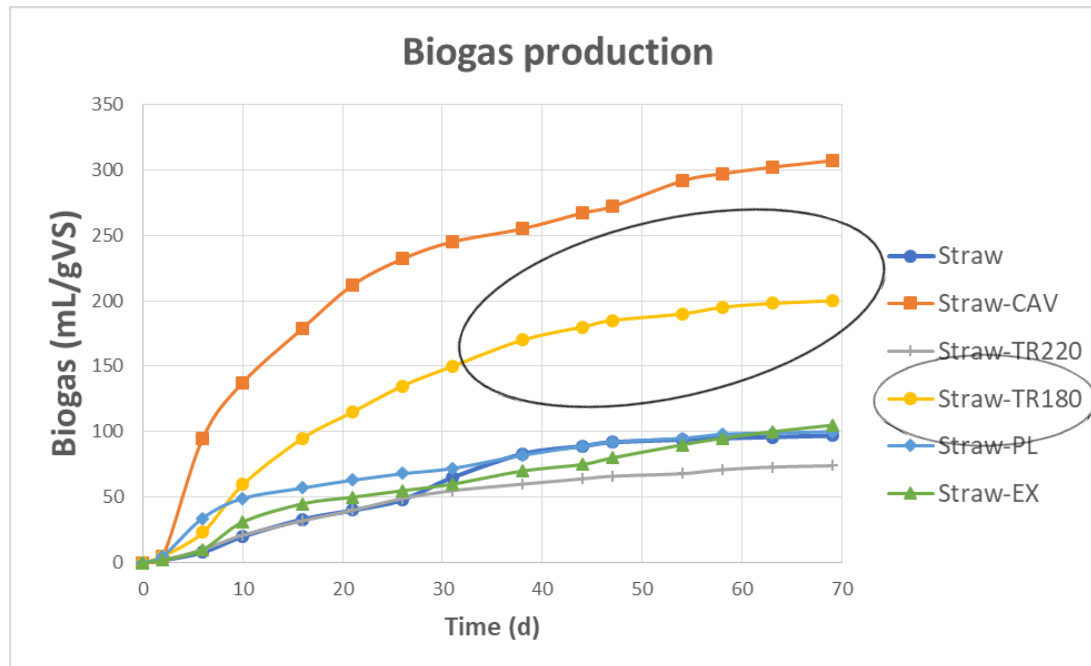




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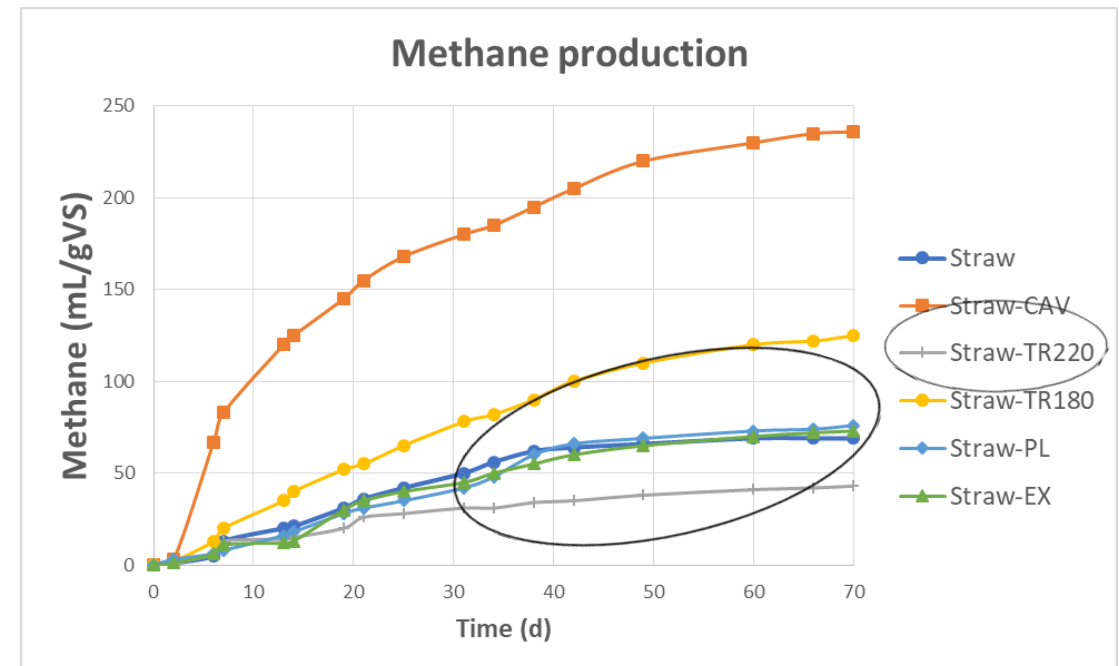
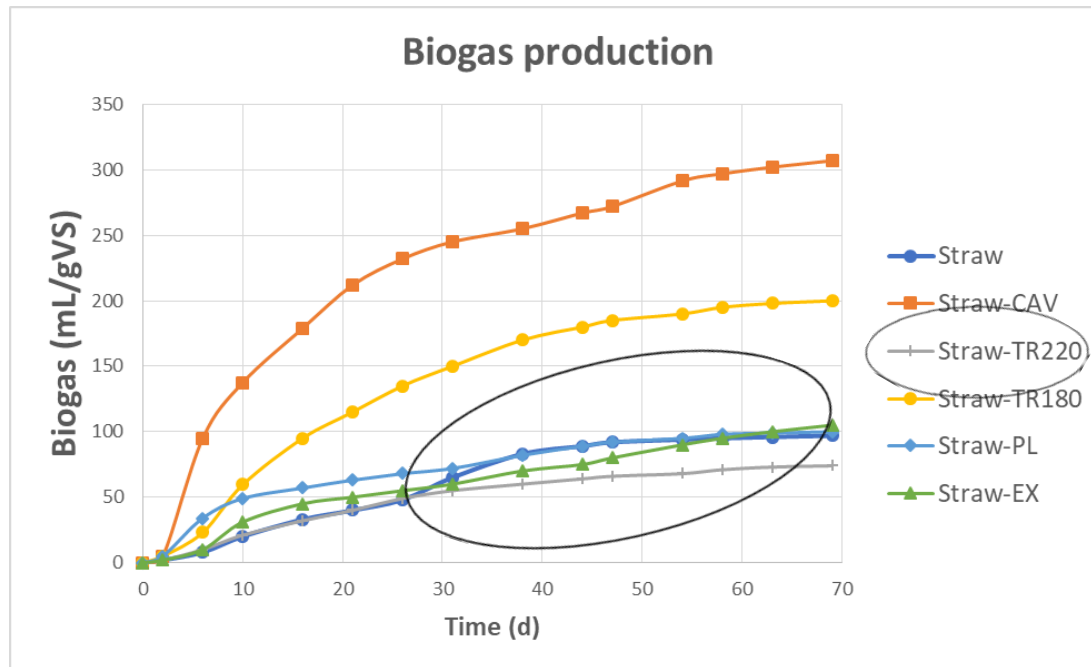
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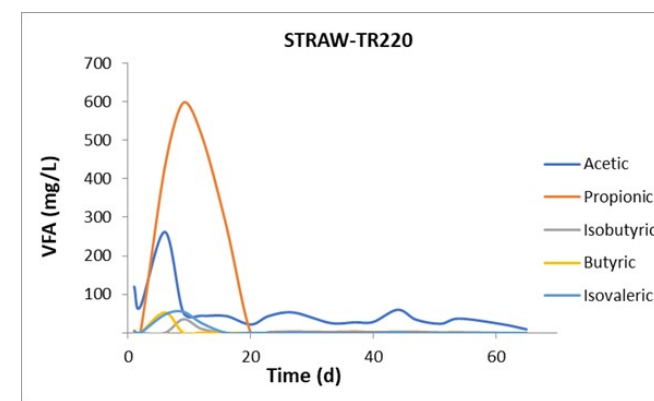
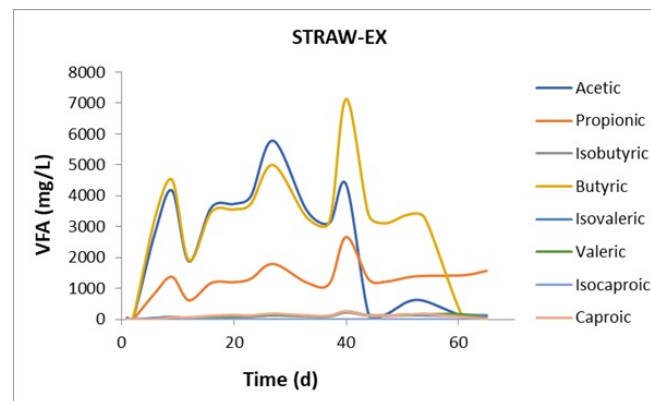
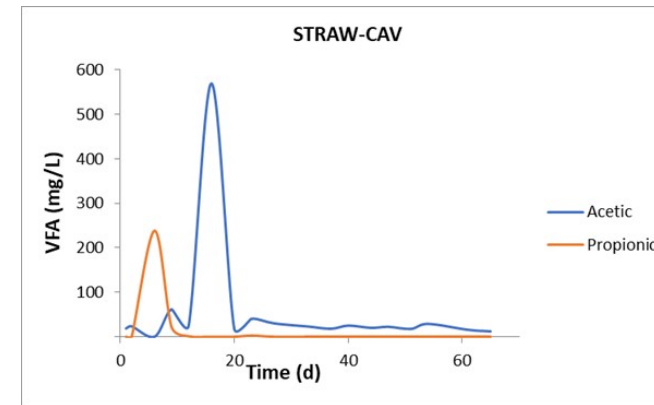
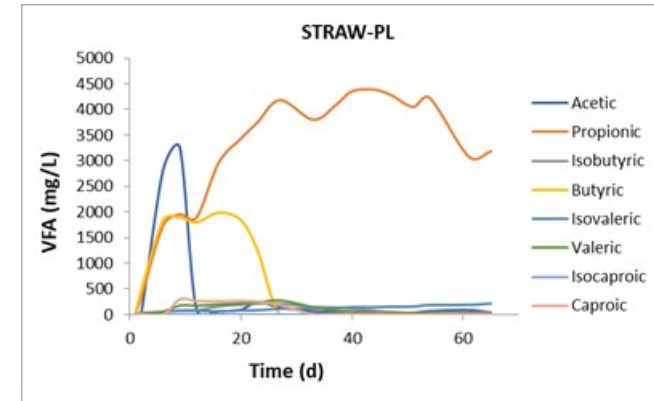
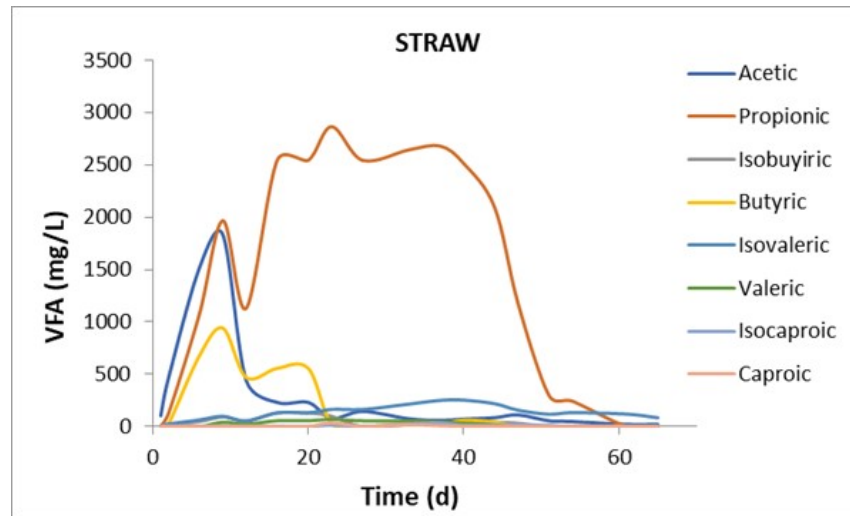
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# First results

**Exp. 3:** Digestion of milled cereal straw  
WWTP digestate (fresh) as inoculum

Volatile Fatty Acids production:

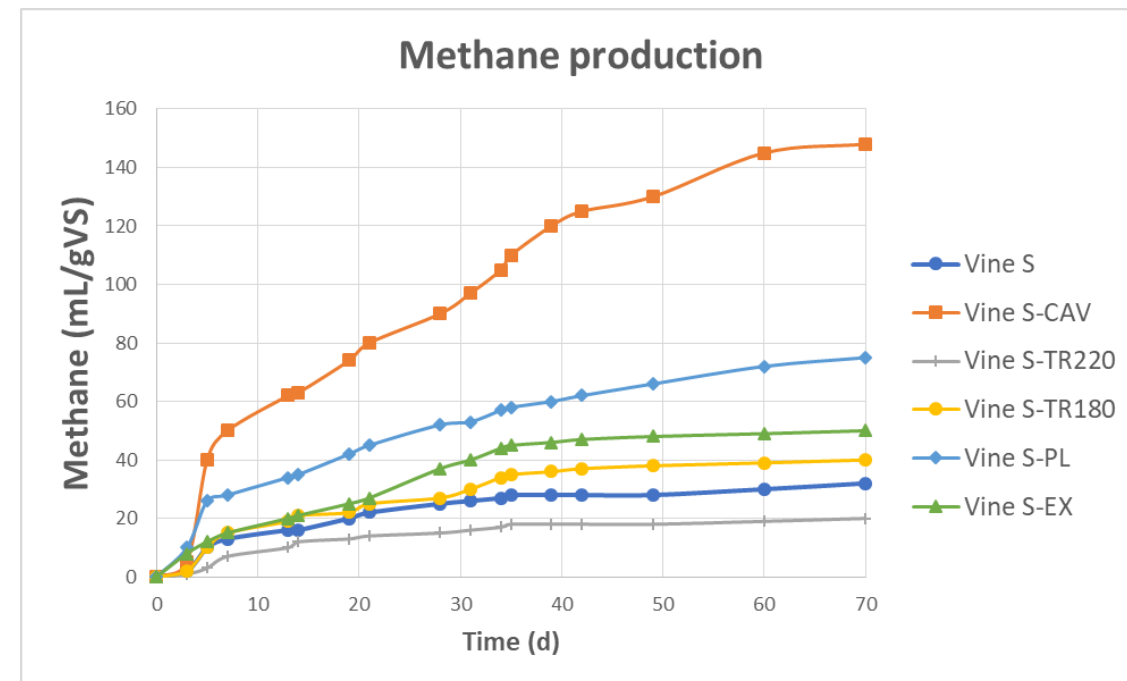
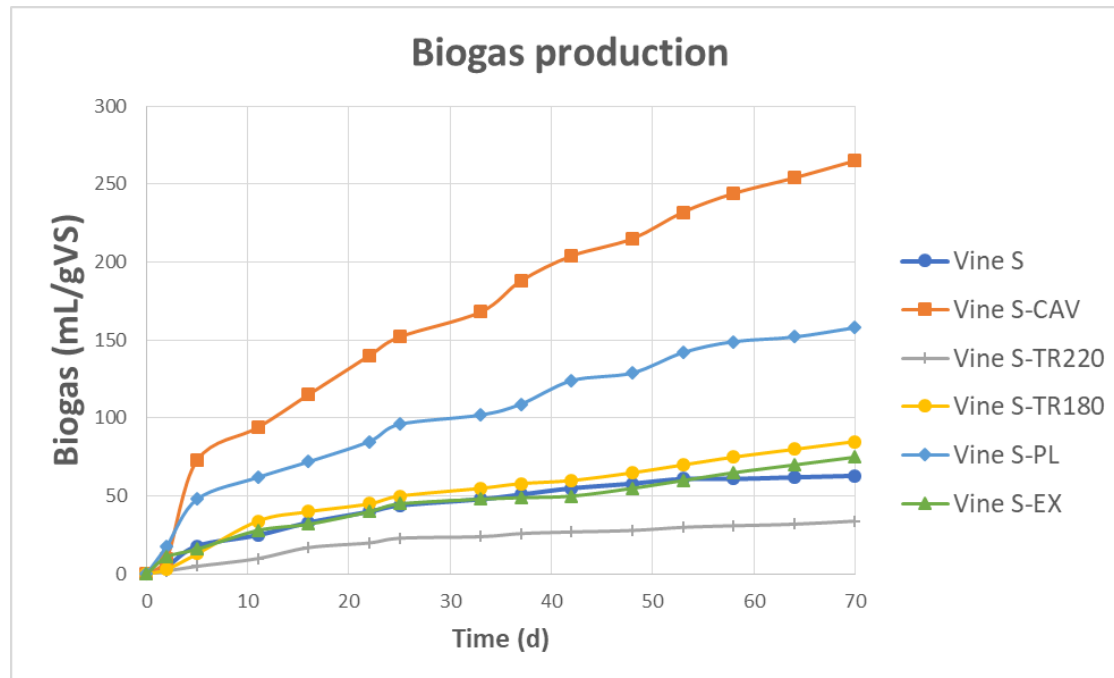




# First results

## Exp. 4: Digestion of milled vine shoot using WWTP digestate as inoculum

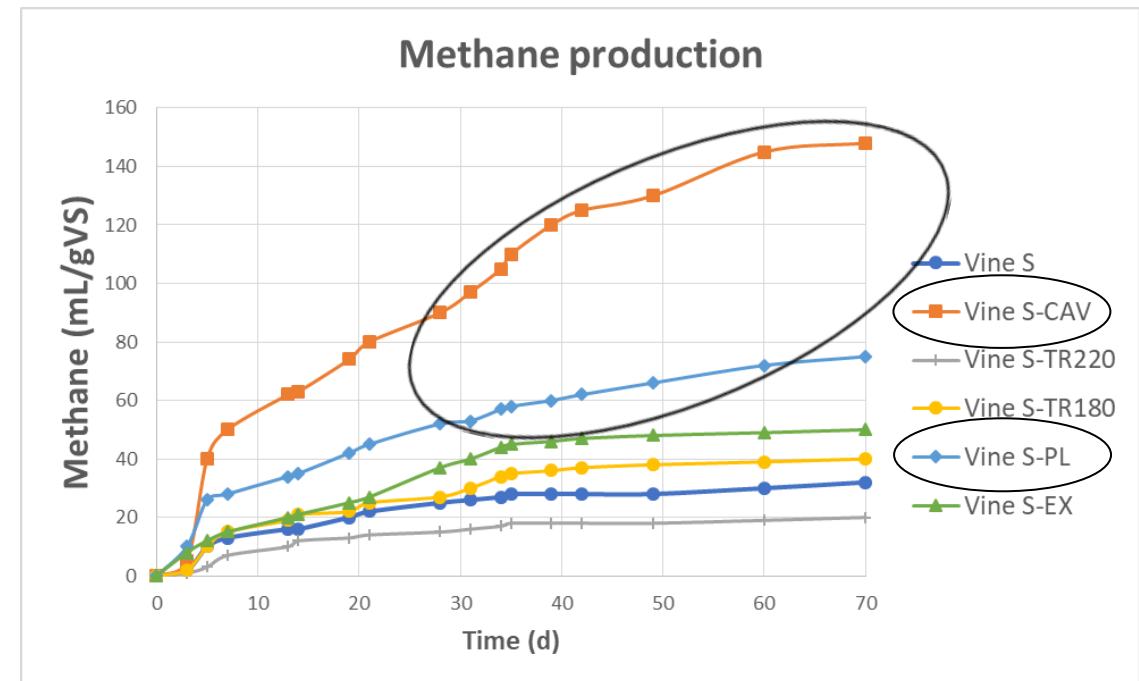
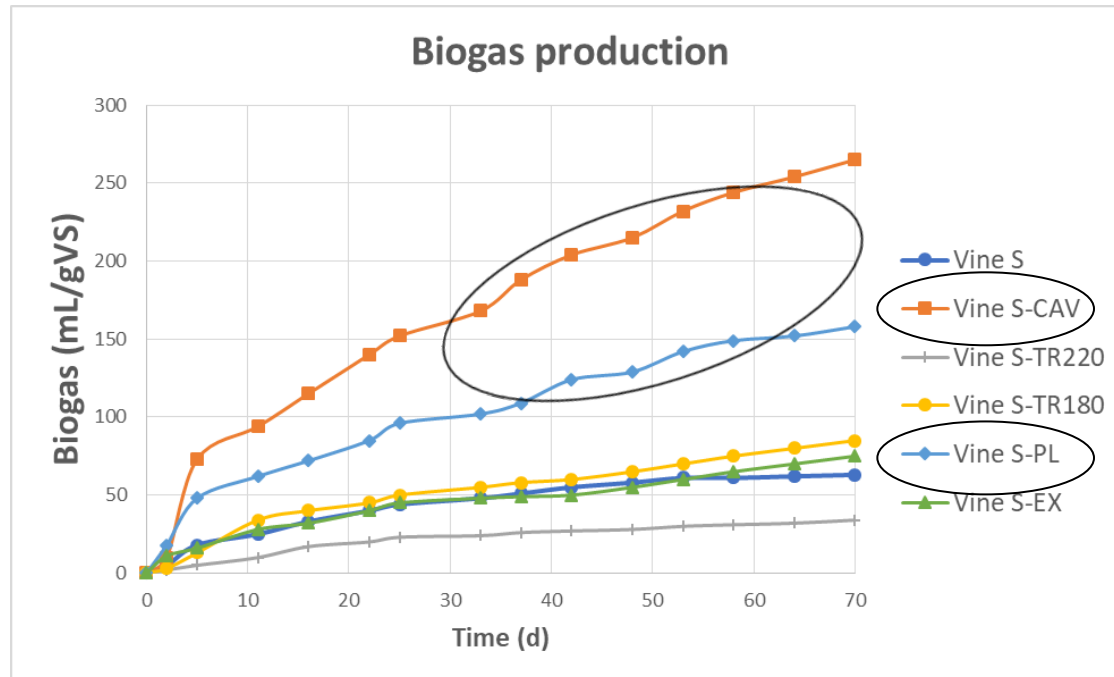
Pretreatments: Cavitation (CAV), Torrefaction at 220°C (TR220), Torrefaction at 180°C (TR180), Pelleting (PL) and Extrusion (EX)



# First results

## Exp. 4: Digestion of milled vine shoot using WWTP digestate as inoculum

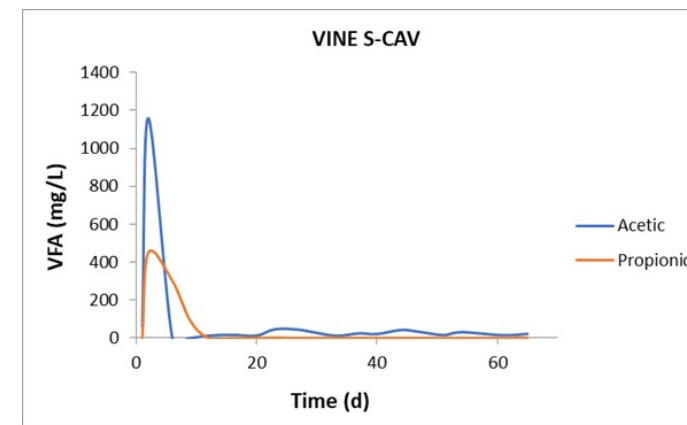
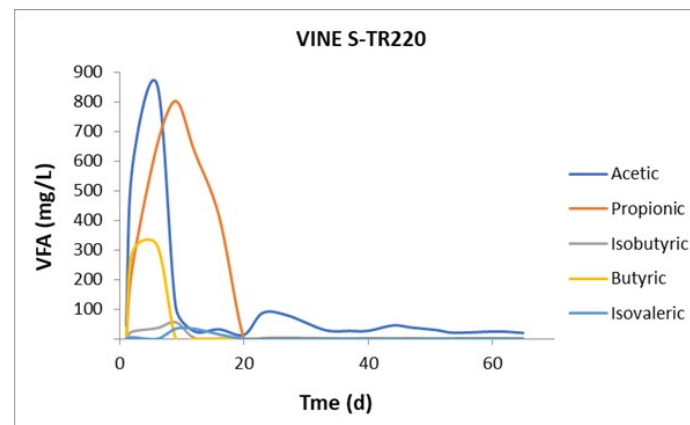
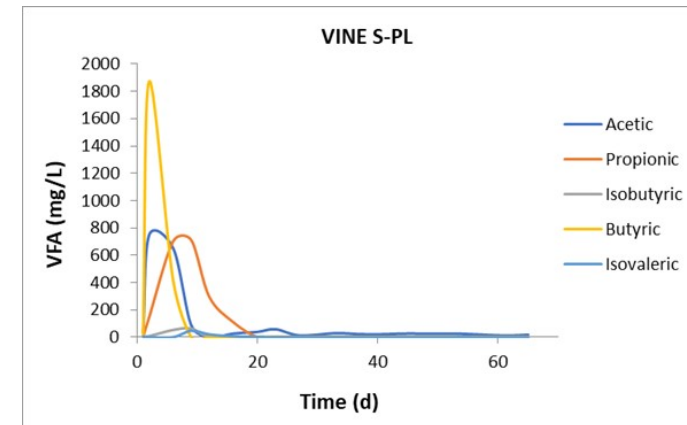
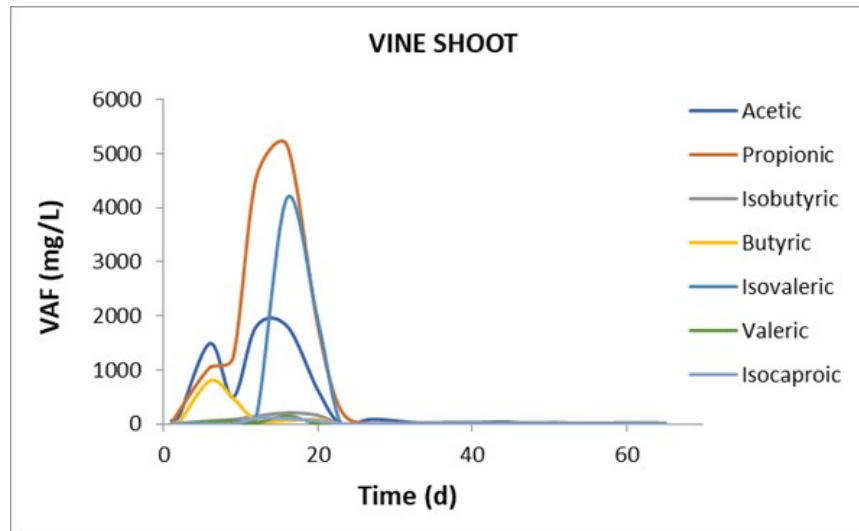
Pretreatments: Cavitation (CAV), Torrefaction at 220°C (TR220), Torrefaction at 180°C (TR180), Pelleting (PL) and Extrusion (EX)



# First results











## Exp. 4: Digestion of milled vine shoot using WWTP digestate as inoculum

### Volatile Fatty Acids production:



# Conclusions (1)

- The pretreatments assayed showed a different degree of effectiveness on methane production when applied to barley straw or to vine shoots.

	Barley straw	Vine shoot
Torrefaction 220°C		
Torrefaction 120°C		
Cavitation		
Pelleting		
Extrusion		



## Conclusions (2)

- The **cavitated** samples exposed to the air shows the formation of mold colonies within 24 hours after the application of the pretreatment. This demonstrates that organic matter is much more accessible to microorganisms than in the unpretreated sample (a fact that would justify its greater biodegradability).
- In the case of **torrefaction**, only low pretreatment temperatures (180°C) favor the subsequent biodegradation of the biomass. High pretreatment temperatures (220°C) inhibit biogas formation.
- The **distribution of individual VFAs** largely depends on the type of biomass and also on the type of pretreatment used.
- A higher peak of acetic at the beginning of the assay seems to promote biogas production.
- High accumulation of VFAs in some test could be related to anaerobic process inhibition.

## Next steps



- Analyse relation between VFA profiles and methane production in relation to the pretreatment selected and the type of biomass.
- Study the influence of torrefaction temperature on biomass biodegradability in the range 100-220 °C.
- Scale-up cavitation pretreatment.
- Analyse economic feasibility of cavitation and torrefaction as biomass pretreatments.

**Thank you for your attention**

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