Torrefaction as a pretreatment of lignocellulosic biomass in anaerobic digestion

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Keywords: pretreatment, lignocellulosic, VFA, biogas.
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Lignocellulosic biomass is very abundant throughout the world and is the focus of many investigations that seek the best method to value this raw material as a source of renewable energy. Among these methods, the anaerobic digestion of lignocellulosic residues is an option that is gaining interest for the production of methane (Paul and Dutta, 2018; Rodionava *et al*., 2021). However, the intrinsic structure of this material, composed mainly of cellulose, hemicellulose and lignin, makes its digestibility difficult and with it, its degradation and conversion into biogas. For this reason, when evaluating the anaerobic treatment of lignocellulosic biomass, it is also necessary to consider a pretreatment stage that weakens the molecular structure of this material and facilitates attack by the methanogenic microbiota (Cai *et al*., 2021).

Physical, chemical or microbiological pretreatment methods can be used to increase the biodigestibility of lignocellulosic residues. Among them, chemical pretreatments have the limitation of producing secondary contamination while biological pretreatments are usually slow and difficult to control. However, physical pretreatments have advantages in terms of ease of implementation at different scales, operational convenience and lower investment (Atelge *et al*., 2020; Zhao *et al*., 2022). Torrefaction is a type of physical pretreatment in which lignocellulosic biomass is subjected to heating at a certain temperature. With this pretreatment, the density of the material, as well as the porosity of the lignocellulosic microstructure can increase considerably, which is convenient, not only in terms of biodegradation, since the connected lignocellulosic structure opens up, potentially making it more accessible to microorganisms, but also for transport and storage, which makes full-scale implementation more feasible (Doddapaneni *et al*., 2018).

Torrefaction temperature is a key parameter that must be carefully selected for optimal results in a subsequent anaerobic digestion process. Too low torrefaction temperature may not substantially affect the structure of the lignocellulosic biomass, and thus not favour the biodegradation process. Too high torrefaction temperature, in addition to having a negative impact on the economy of the global process, could generate secondary products that affect the operability of the microbiota, and generate an inhibition of the subsequent anaerobic digestion process. On the other hand, the composition of the lignocellulosic biomass to be digested is also a factor to take into account when selecting the conditions for the torrefaction stage (Hidalgo *et al*., 2021).

The objective of this study is to investigate the effects of the torrefaction pretreatment temperature on the anaerobic digestion process, in terms of biogas production and generation of volatile fatty acids, when two feedstocks with different content of lignin, cellulose and hemicellulose, such as barley straw and vine shoots, are subjected to these processes consecutively.

The barley straw and the vine shoot were first mechanically milled, using a blade mill and a hammer mill respectively, so that they reached a particle size of 2 mm. Then milled biomass samples were torrefacted in batch mode at 100, 150, 180 and 200 °C for 30 min. The ability of the different temperatures to create structural damages on the biomass morphology was analysed by SEM. In order to study the biodegradability of the pretreated biomass, batch experiments were run in glass serum bottles with a liquid volume of 500 mL (1000 mL of total volume). All the experiments were carried out at 34±1 ºC in a thermostatic room. Digestate from an anaerobic reactor operating in a municipal wastewater treatment plant, with a VS concentration of 10,0±0,5 g L^{-1}, was used as inoculum for the anaerobic test. Final inoculum concentration in the tests medium was 7.0±0.5 g L^{-1}. The substrate/inoculum (S/X) ratio was maintained for all samples at 4.2±0.4 gSVsubstrate / gSVinoculum. Biogas production was automatically measured by using pressure transmitters (Desin Instruments, TPR-14 / N, range 0-1 bar) connected in the upper space of each reactor. The biogas composition was measured using a Varian CP-4900 Micro-GC chromatograph with a Thermal Conductivity Detector. The individual Volatile Fatty Acids (i.e., acetic, propionic, isobutyric, butyric, isovaleric, valeric acids and others) were quantified using GC equipped with flame ionization detector (GC-FID) (GC2014, Shimadzu, Japan). Biomass composition, in terms of main lignocellulosic fractions contents (cellulose, hemicellulose and lignin) was determined by thermogravimetric analysis and pseudocomponent kinetic model as detailed in Díez *et al*. (2020).

It is first observed that the torrefaction temperature has an influence on the resulting composition of the pretreated samples, as shown in Table 1.

Table 1. Composition of biomass samples after torrefaction at different temperatures.

Note: these results are preliminary and will be confirmed in the full version of this paper.

	Torrefaction temperature

Barley straw	untreated	100 ºC	150 ºC	180 ºC	200 ºC
Cellulose	51,6%	51,4%	*48,6%*	52,9%	53,3%
Hemicellulose	31,4%	31,4%	*32,9%*	29,6%	28,8%
Lignin	17,0%	17,3%	*18,5%*	17,5%	17,9%
Weight loss	0	1,41%	*8,25%*	2,97%	4,68%
Vine shoots	untreated	100 ºC	150 ºC	180 ºC	200 ºC
Cellulose	23,2%	22,2%	*19,5%*	*20,9%*	22,3%
Hemicellulose	49,2%	48,6%	*49,6%*	*50,7%*	47,4%
Lignin	27,5%	29,3%	*31,0%*	*28,4%*	30,3%
Weight loss	0	6,05%	*11,14%*	*3,04%*	9,28%

For both feedstocks the results (trends) suggest that, as the torrefaction temperature increases, the relative percentage of hemicellulose in the samples decreases, while cellulose content increases for barley straw and decreases for vine shoots. Relative content in lignin increases in both cases. Also in both cases a global weight loss is observed, although this loss is more significant in the case of the vine shoot.

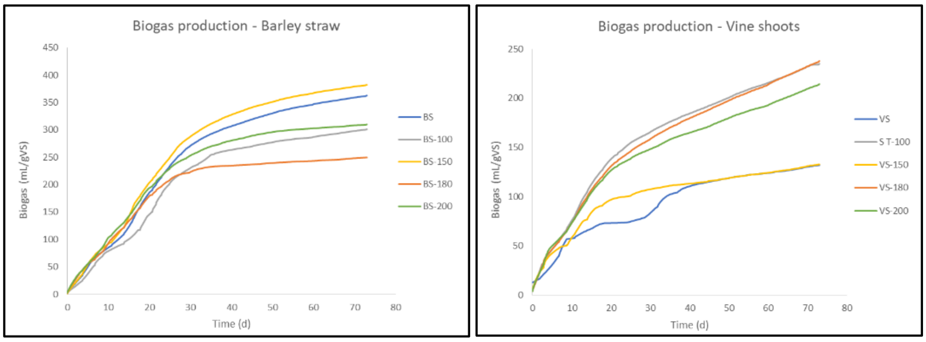

Figure 1. Biogas production.

The pretreatment assayed (Figure 1) showed a different degree of effectiveness on biogas production according the test temperature when applied to barley straw or vine shoots. In both cases, the application of the pretreatment revealed to affect positively or negatively the biogas yield depending on the specific pretreatment temperatures. Another result observed is the distribution of individual volatile fatty acids (VFA) is strongly depended on the type of biomass and also on the pretreatment temperature. During the anaerobic digestion of untreated barley straw or vine hoots, propionic and acetic acids dominated the VFAs profile. These acids are also predominant when pretreated samples are digested but production profiles change drastically what clearly influences the biodegradation curves.

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