Hydrothermal Pretreatment of sugar beet residues targeting sugars production

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Sugar beets (along with the crops residues) are lignocellulosic materials, which are three-dimensional bio-polymers comprised of sugars (cellulose and hemicellulose) and lignin. The sugars contained in the biomass can be valorized as nutrients in enzymatic hydrolysis. However, pre-treatment of biomass is necessary so that the lignin structure can be distracted in order to reach the holocellulose. A sustainable process (low cost and eco-friendly) to produce oligomeric and monomeric sugars is hydrothermal pretreatment. The process utilizes hot water properties, so that hemicellulose and secondarily cellulose can be extracted as sugars in water. The main parameters affecting the process yield are temperature and residence time. The degradation reactions occurring are based on autogenous hydrolysis by valorizing acetic acid originated from hemicellulose for cracking reactions of polysaccharides to oligo and mono-saccharides[1].

In this study, the hydrothermal pre-treatment of sugar beet residues was examined at low temperature range of 110° to 160°C for 15 – 30 min in order to produce sugars for enzymatic hydrolysis. Also, a comparative study was conducted between hydrothermal pretreatment and acid hydrolysis at same conditions by using sulfuric acid aqueous solution at 1 wt.% concentration. The two products (liquid product and the solid residue) were analyzed via HPLC to determine the type and quantity of the sugars produced. The results indicated that acid hydrolysis leads to enhanced sugars production compared to hydrothermal pre-treatment at this range of parameters. Also, the liquid product in acid treatment has higher concentration of diluted sugars, significant concentration of glucose (derived from cellulose) and lower proportion of insoluble hemicellulose. Besides that, solid residue is less in acid treatment and contains lower amounts of hemicellulose and glucans (cellulose structure molecule).

Finally, as for the examined parameters, there is similar trend in both methods. Increment in temperature to 135°C leads to enhanced production of glucose and sugars derived from hemicellulose, while insoluble hemicellulose concentration is decreased. On the other hand, further increase in temperature gives lower sugars yield (especially in hemicellulose) due to further reactions of sugars into furfurals which are unwanted as they act as enzyme inhibitors. So, the optimal set of parameters were 135°C and 30 min in acid treatment. This set of conditions produces 0,32g of glucose per 10g of biomass and 0,78g of sugars originated from hemicellulose (max yield of 69% in saccharification of hemicellulose).

The experiments were conducted at the Centre for Research & Technology Hellas (CERTH) in a bench top, high-pressure stirred batch reactor with internal vessel volume of 250 mL (Parr 4576A). The reactor is coupled with a J type thermowell for heating and a U type cooling coil for rapid temperature dropping. In a typical run, the reactor is loaded with 10 g of feedstock and 100 mL of deionized water (solid to liquid ratio of 1/10). Then, the reactor is sealed and purged 3 times with nitrogen to remove air. Finally, the reactor inlets are compressed to 20 bars with nitrogen (to keep liquid state), then heated and kept to the desired temperature according to set of conditions before cooldown.

Pretreatment of biomass renders solid, liquid and gas products. Gas products were not analyzed in this study. The separation of the products starts with vacuum filtration of the mix in a Buchner funnel with filter paper. The liquid collected is the aqueous phase product, containing the solvent with liquified sugars and some lignin. The solids are dried and weighted, while the liquid product is ready for HPLC analysis. The HPLC analysis was conducted via a SHIMADZU UFLC XR equipped with a Shodex SP0810 column and a Shimadzu RI detector. The process is depicted in the following figure 1.

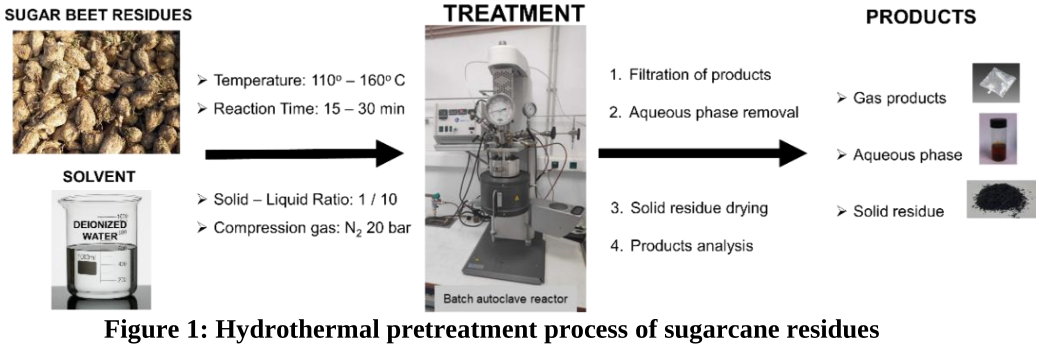

Figure 1: Hydrothermal pretreatment process of sugarcane residues

The solid residue quantity and composition in each condition indicates the trend in components dilution (Figure 2). So, the results show a large reduction of cellulose and hemicellulose (over 50 wt.%) at solid products. Also, lignin content is not reduced in solid residue which is desirable as lignin mitigation to the liquid product could inhibit enzymes functionality. Moreover, by comparing the different sets of parameters, increased temperatures reduce all structural components in residue which means there is significant mitigation in liquid product.

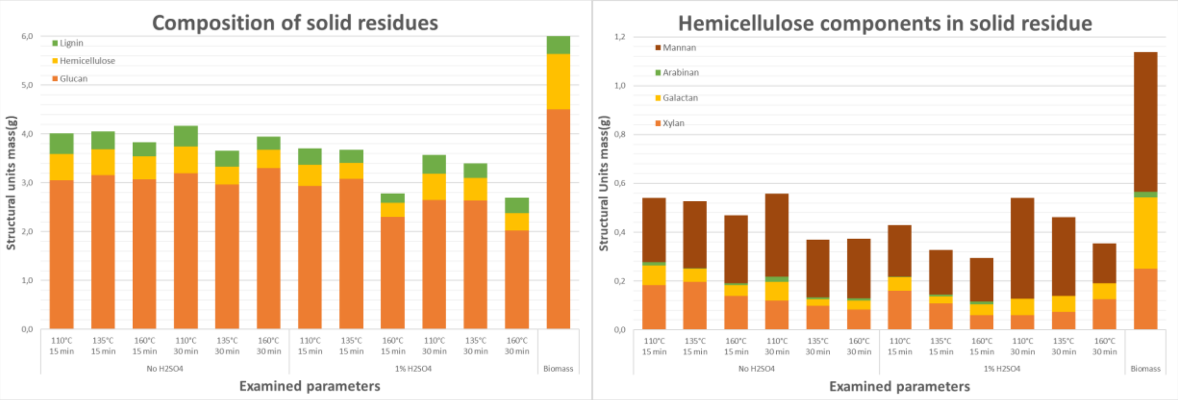

Figure 2: Structural composition and hemicellulose components of solid residues

The analysis on sugars derived from hemicellulose in solid residue showed saccharification yield over 50% in some cases (Figure 2). So, at more severe conditions, hemicellulose is easier converted to oligomer and monomer sugars. Arabinan can be completely diluted in aqueous phase, galactan is converted in maximum yield of 70 wt% while mannan production can vary depending on set of conditions and the maximum yield (72%) is accomplished at acid environment and more severe conditions.

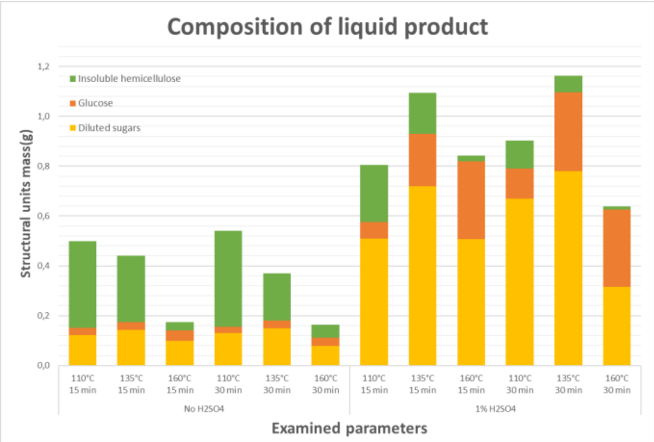

Figure 3: Composition of aqueous phase products

On the other hand, although hemicellulose decomposition occurs at higher temperature and time, this trend is not observed in the analysis of the aqueous phase which is the main product (Figure 3). In hydrothermal pretreatment, sugar yield is poor in each set of parameters and the most suitable set of parameters are 135°C and 30 min. Instead, at more severe conditions, sugar yield is lower. In acid treatment this trend is even more discrete as sugars yield is significantly higher. So, although at high severity, hemicellulose saccharification occurs at higher yields, it appears that xylose is secondarily converted to furfurals and hexoses are converted to HMF (5-hydroxymethyl furfural). Furfurals act as inhibitors in enzyme functions and they are unwanted. So, this reaction pathway is the reason why the optimal and more suitable conditions are achieved in average and no at maximum temperature.

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