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## High-intensity ultrasound and hydrodynamic cavitation as powerful treatment for biomass conversion

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### 1. Introduction

Agro-industry residues generated as wastes during or after processing of agricultural crops are the main resource of lignocellulosic feedstocks available in huge amounts. Delignification is a primary step for further biomass fermentation to biofuels or conversion to fine chemicals. High-intensity ultrasound<sup>1</sup> (US) and hydrodynamic cavitation<sup>2</sup> (HC) have been used as powerful pretreatment for a full valorization of biomass. Due to their peculiar mechanisms, cavitation treatments give effective lignocellulosic matrix dismantling and delignification at low temperature (35-50°C). Cavitation also causes decrystallization of cellulose due to partial depolymerization. Within the H2020 project US4GreenChem starting from lab scale investigation, new protocols and equipment features have been designed for lignocellulosic biomass pre-treatment, with the aim to enhance the efficiency of enzymatic hydrolysis of cellulose and lignin recovery.

### 2. Experimental

Wheat straw (*Triticum aestivium*) was used as raw material (original lignin content 21%, and particle size <0.2) for both pre-treatments:

1) HC *pre-treatment* (30 min) was performed in a rotor-stator reactor ROTOCAV (E-PIC S.r.l.) that works in loop mode at 3000 rpm, with 0.4 Kg of raw material. The pre-treatment was performed at 50°C in NaOH solutions (5-10 wt% on dry biomass) with solid/liquid ratio = 1:50.

2) *High-intensity US pre-treatment* (30, 60, 90, 120 min) was performed in a multi-frequency reactor (WEBER Ultrasonics GmbH) (working both in batch and flow mode) using different frequencies (25, 80 or 120 kHz). The pre-treatment was performed at 35°C treating from 2 g to 2 kg of wheat straw in NaOH solutions (10 wt% on dry biomass) with solid/liquid ratio = 1:20.

The pre-treated mixtures were filtered and both liquid and solid fractions were recovered after neutralization. Analysis of individual phenolic compounds on liquid fractions were performed on ACQUITY UPLC H-Class system coupled to SYNAPT G2-Si High Definition Mass Spectrometer.

### 3. Results and discussion

Because of its high content of cellulose, wheat straw is a good raw material for ethanol production. For the bioconversion of lignocellulosic materials to ethanol, pretreatment of the material prior to enzymatic hydrolysis is essential to obtain high overall yields of sugar and ethanol.<sup>3</sup> In this work, both US and HC treatments showed an ability to effectively disrupt the lignocellulosic matrix of wheat straw and maximize sugars yields in the following enzymatic hydrolysis performed with improved enzyme pools at Teknologian

Tutkimuskeskus (VTT, Finland) and UAB Biocentras (Lithuania). A sequential combined treatment with HC and flow US could reduce the overall treatment time. Preliminary results showed that cavitation pretreatments carried out with suitable equipment and optimized conditions are competitive with classic steam explosion process. US and HC can be easily scaled up enabling loop or flow-mode processes (see Figure 1). The composition of wheat straw aqueous extracts obtained by treatments with US and/or HC aimed at lignocellulose matrix disintegration was investigated. These treatments developed in the H2020 project US4GreenChem are the first step of a novel biorefinery process that dramatically enhances efficiency of enzymatic hydrolysis of cellulose. A full chemical profile characterization of liquid fraction after wheat straw pre-treatment was performed by UHPLC – ESI-MS/MS methods. This fraction contains several phenolic compounds (52 -120 mg of GAE per g of oven dried sample) which can be considered as value-added products. Moreover, all fractions possess good antioxidant properties (1848-3574 Trolox\* equivalent, mmol/g).



**Figure 1.** HC (left) and high-intensity US reactors (right) for wheat straw pre-treatment.

#### 4. Conclusions

The present work has clearly established the important effect acoustic and hydrodynamic cavitation for the effective treatment of wheat straw in alkaline conditions. The potential economic and environmental impacts of combined technologies will be evaluated for a full valorization of lignocellulosic biomass and the bioconversion to ethanol.

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