Aqueous ammonia soaking pretreatment of spent coffee grounds for enhanced enzymatic hydrolysis

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Keywords: aqueous ammonia soaking, pretreatment, enzymatic hydrolysis, spent coffee grounds Presenting author email: panag.tsafrak@gmail.com

Efficient utilization of biomass is a pillar of the bio-economy, aiming at the sustainable use of feedstocks for the production of new materials. In this context, the abundant waste stream of Spent Coffee Grounds (SCG), a lignocellulosic waste material obtained from the retail coffee outlets, could be considered as a valuable biopolymer resource for environmentally friendly product design.

Coffee beans are composed mainly of polysaccharides and more specifically hemicelluloses (mannans, galactomannans, arabinogalactans) and cellulose (Tian et al., 2017). The polysaccharides that are not water extractable during coffee beverage preparation, remain in the residual material. Therefore, SCG could be used as a source to obtain fermentable sugars, which could be subsequently valorized through different bioprocessing routes. Several studies have been published regarding the use of SCG for the biotechnological production of polyhydroxyalkanoates (Obruca et al., 2015), carotenoids (Petrik et al., 2014), and bioethanol (Kwon et al., 2013). However, the composite lignocellulosic structure makes this biomass recalcitrant to biological and chemical biotransformations and the downstream processing difficult, and thus necessitates the use of diverse pretreatments and conversion methods to make it more amenable to further processing and valorization (Williams et al., 2016). Among these methods, the most common ones are acid hydrolysis with H₂SO₄ and/or enzymatic hydrolysis with the use of hydrolases, i.e. microbial mannanase and/or cellulase preparations (Juarez et al., 2018; Mussato et al., 2011). However, these methods do not lack drawbacks since acids are highly corrosive chemicals and require large amounts of water to separate degraded cell wall components, whereas the enzymatic methods are more costly, require specific conditions and are less effective in biodegrading lignocellulosic residues.

Aqueous Ammonia Soaking (AAS) is lately considered as an efficient alternative pretreatment method for lignocellulosic feedstocks (Kim et al., 2007; Mouthier et al., 2019), since it removes lignin selectively and retains both hexose- and pentose-based carbohydrates (Kim et al., 2016). In addition, ammonia can be recovered and recycled, has low cost, and is widely used in chemical industries; moreover, AAS has low energy requirements (Mouthier et al., 2019). In the present study, aqueous ammonia soaking was examined as a potential pretreatment stage for any subsequent bioconversion of SCG, as it cleaves ether and ester linkages in lignin-carbohydrate complexes and thereby constitutes an effective swelling reagent for lignocellulosic biomass by interrupting the structural integrity of lignified plant cell wall residues. To explore this process more holistically, the objective of the current work was to design an efficient pretreatment with this alkali reagent for the recovery of hemicelluloses and cellulose fragments from SCG, in order to use them as potential carbon sources for microbial fermentation applications.

Triplicate experiments were conducted in 100 ml Duran laboratory bottles with 50 ml ammonia solution (28-30% v/v). The studied parameters were treatment time (0.5-6 h), SCG solids loading (5-20% w/v), and temperature (40-80°C). Each parameter was studied individually while keeping the other parameters constant. Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and Scanning Electron Microscope (SEM) of the samples before and after pretreatment were performed in order to investigate compositional and morphological changes of the biomass structures. Following the AAS, the pretreated SCG was subjected to enzymatic hydrolysis using commercial cellulases and hemicellulases at constant loadings. Reducing sugars and other degradation products obtained from the pretreated and saccharified samples were measured by HPLC.

One-factor-per-time method with successive variations in examined variables, as adopted herein, is the first step for a subsequent optimization of the entire process. In this context, it must be pointed that the efficiency of any pretreatment method largely depends on many process parameters, including the nature (composition and structural features) of the examined biomass residue. Therefore, it is essential to study the impact of each parameter separately, prior to the investigation of potential interaction effects for each plant residue. To the best of our knowledge, the AAS method has not been previously used for pretreatment of SCG, and thus the present work is setting the ground for the development of an effective, affordable and eco-friendly processing scheme for the valorization of this sugar rich biomass waste material.

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Acknowledgments

«This research has been co-financed by the European Regional Development Fund of the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH – CREATE – INNOVATE (project code: T2EDK-02242)».