



Optimizing deep eutectic solvent pretreatment for enhanced glucan recovery from miscanthus

Patricia J. Piedade,^a Michał M. Nowotarski,^b Gabriela Dudek^b and Rafal M. Lukasik^{b,*ac}

This work focuses on the use of the deep eutectic solvent composed of choline chloride and acetic acid (1:2) in the pretreatment of the perennial energy crop *Miscanthus*, commonly called silvergrass. The pretreatment time and temperature were optimized to achieve a maximum of the glucan and minimum of the lignin contents in the pretreated biomass. In the optimization approach, the maximal glucan recovery in the pretreated solid was also considered. The performed optimization resulted in conditions (2 h 52 min and 150 °C), at which a pretreated biomass contained 74.1 wt% and 9.5 wt% of glucan and lignin respectively, and a glucan recovery was as high as 87.0 wt%. Furthermore, the biocompatibility of deep eutectic solvent was evaluated by using enzymatic hydrolysis washed and unwashed pretreated biomass produced at optimal conditions. The enzymatic hydrolysis of washed biomass resulted in higher glucan and xylan conversion than those achieved from unwashed biomass, deeming the step of biomass washing necessary. This was confirmed by the fractal kinetics modelling that confirmed higher accessibility of glucan for washed biomass than for unwashed *Miscanthus* sample.

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1. Introduction

The exponential growth of the global population and the expansion of energy-intensive industries have resulted in the unsustainable overexploitation of our limited resources. While fossil fuels have propelled remarkable industrial advancements, their utilization comes at a considerable environmental cost. The combustion of these non-renewable resources has led to the emission of substantial amounts of greenhouse gases into the atmosphere, deepening global warming and climate change.¹ Therefore, it is necessary for industries to move towards sustainable alternatives. In this context, lignocellulosic biomass emerges as a promising solution.² Derived from non-edible organic sources such as crop residues, forestry waste, and dedicated energy crops, biomass offers a renewable and carbon-neutral resource that can be effectively exploited to generate a variety of commodities including, *e.g.*, biomaterials and biofuels.³

Utilizing agricultural and forestry biomass residues is important to limit potential greenhouse gas emissions as well as landfill waste. Hence, an integration of energy crops is

necessary to address the drawbacks of first-generation biomass valorization.⁴ Energy crops can be a controlled and reliable source of feedstock that can counter the seasonality issues typical for agricultural residues. The combination of these feedstocks in the context of biorefinery allows for more controlled and continuous production of renewable resources. Additionally, the use of underutilized degraded or marginal lands can ensure a sustainable and efficient approach for sustainable alternative to fossil resources.⁵

Miscanthus has been widely studied for its potential as an energy crop due to its high biomass productivity, low need for pesticides, fertilizers, and low water footprint.⁶ However, the conversion of *Miscanthus*, like any lignocellulosic biomass, presents significant challenges, mainly due to its resistant nature.⁷ Lignocellulosic biomass requires pretreatment before conversion, particularly in biological conversion processes, to make the cellulose more accessible to enzymatic hydrolysis.⁸ This pretreatment step is one of the most costly and energy-intensive stages of the overall biomass conversion process.^{9,10} Furthermore, conventional volatile organic solvents used in pretreatment, although highly efficient, present concerns regarding their toxicity and harmful environmental impact.^{11,12}

Deep eutectic solvents (DES) have emerged as a promising alternative to traditional solvents, offering various potential economic and environmental benefits.^{12–14} DES are solvents with low volatility and toxicity, and are easily prepared by mixing a hydrogen bond acceptor (HBA), usually choline

^a Laboratório Nacional de Energia e Geologia I.P., Unidade de Bioenergia e Biorrefinarias, Estrada Do Paço Do Lumiar 22, 1649-038, Lisboa, Portugal.
E-mail: rafal.lukasik@lukasiewicz.gov.pl

^b Department of Physical Chemistry and Technology of Polymers, Silesian University of Technology, Strzody 9, 44-100 Gliwice, Poland

^c Łukasiewicz Centre, Łukasiewicz Research Network, 19 Poleczki str., 02-822 Warsaw, Poland