

# Design of green routes for cellulose extraction from a sugarcane by-product

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## Introduction

Cellulose, the world's most abundant natural polymer and a very promising renewable and biodegradable material, can be used to produce valuable cellulose-based products [1]. In the context of sustainable development, lignocellulosic biomass from industrial and agricultural wastes have attracted much attention as cellulose sources [2]. Among agricultural crops, sugarcane plantations are famous for their volumes and large amounts of residues. Sugarcane bagasse (SCB) is the main residue of the sugarcane industry, representing almost 30% of the sugarcane agricultural product [3]. Currently, it is burned or used for low-value applications, so the development of new cellulose-based added-value products is an excellent route to promote its valorization, being of extreme importance for both economic and environmental reasons. A range of physical, chemical and biological methods for cellulose isolation have been proposed [4]. Promising green extraction methods that have recently attracted considerable attention include the use of deep eutectic solvents (DES) [5] and autohydrolysis (AH) [6].

## Objectives

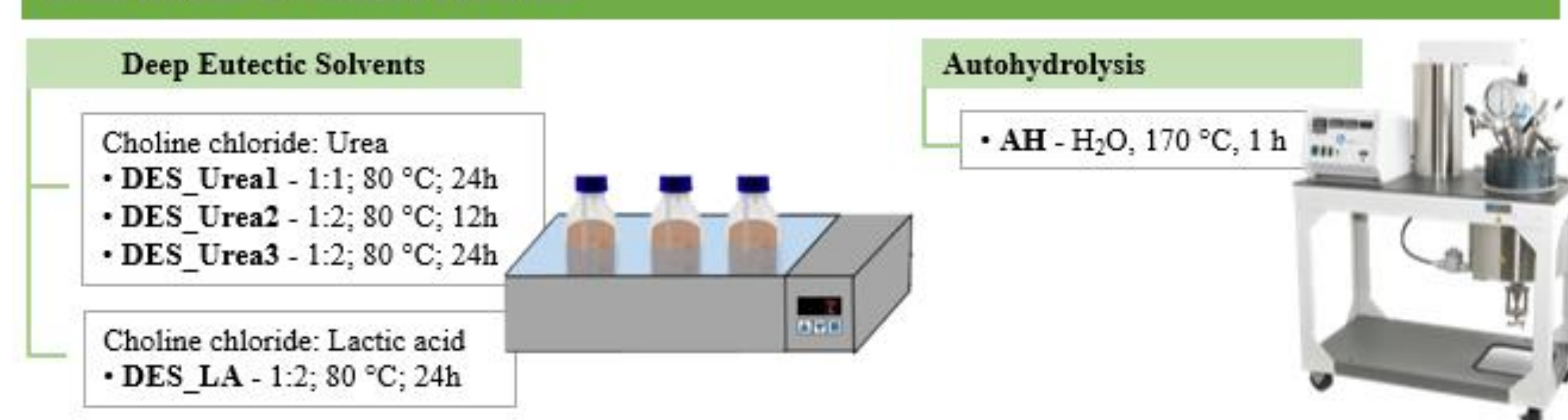
The aim of this work was to obtain cellulose rich fractions from SCB through a green yet effective process. The green methods tested include cellulose extraction using deep eutectic solvents (DES: choline chloride/urea and choline chloride/lactic acid) and autohydrolysis (AH). The impact of the different treatments was evaluated regarding cellulose content, lignin and hemicellulose removal and product yield. Structural characterization were assessed by Fourier-Transform Infrared spectroscopy (FT-IR), Powder X-Ray Diffraction (PXRD) and Scanning Electron Microscopy (SEM) analysis.

## Methods

### Raw material and Cellulose Extraction



### Green Methods for Cellulose Extraction



### Chemical and Structural characterization

The solid fractions obtained were characterized in terms of lignocellulosic composition following the analytical procedures for standard biomass analysis by the National Renewable Energy Laboratory (NREL) [7]. • The FT-IR spectra were recorded using the Frontier™ MIR/FIR spectrometer from PerkinElmer in a scanning range of 550-4000 cm<sup>-1</sup> for 16 scans at a spectral resolution of 4 cm<sup>-1</sup>. • The structure and crystallinity of the samples was evaluated by PXRD using a Rigaku MiniFlex 600 diffractometer with Cu K $\alpha$  radiation, with a voltage of 40 kV and a current of 15 mA ( $3 \leq 2\theta \leq 60$ ; step of 0.01 and speed rate of 3.0° /min). • SEM analysis was performed on a JSM-5600 LV Scanning Electron Microscope from JEOL, Japan. Prior to analysis, the samples were placed in observation stubs (covered with double-sided adhesive carbon tape (NEM tape, Nisshin, Japan) and coated with Au/Pd using a Sputter Coater (Polaron, Bad Schwalbach, Germany). All observations were performed in high-vacuum with an acceleration voltage of 20 kV, at a working distance of 10-11 mm and a spot-size of 4. • Product yield (%) was measured for each experiment and was expressed as the ratio of the mass obtained after the isolation process and the mass of the initial biomass material used.

## Results

The yield and chemical composition of the solid fractions rich in cellulose obtained from the different treatments are presented in Table 1, while its chemical profile, crystallinity and morphology characterized by FT-IR and PXRD (Figure 1a and b, respectively), and SEM (Figure 2) were compared to SCB biomass and standard cellulose from Sigma-Aldrich.

Table 1. Yield and chemical composition of the obtained solid fractions rich in cellulose, as well as the initial SCB

	Yield (%)	Chemical Composition (%)			
		Cellulose	Hemicellulose	Soluble Lignin	Klason Lignin
SCB	-	36.2 ± 1.1	22.6 ± 0.6	4.1 ± 0.2	20.0 ± 0.3
DES_Urea1	90.5	27.8 ± 0.3	20.6 ± 0.4	9.2 ± 0.2	10.9 ± 0.4
DES_Urea2	91.5	30.3 ± 0.3	21.1 ± 0.5	9.6 ± 0.4	11.9 ± 1.1
DES_Urea3	92.7	34.3 ± 1.0	25.9 ± 1.8	11.4 ± 0.3	15.5 ± 1.0
DES_LA	90.7	34.7 ± 0.9	17.7 ± 0.3	5.9 ± 0.2	15.8 ± 0.1
AH	56.2	59.1 ± 0.9	11.4 ± 0.2	2.3 ± 0.6	26.0 ± 0.0

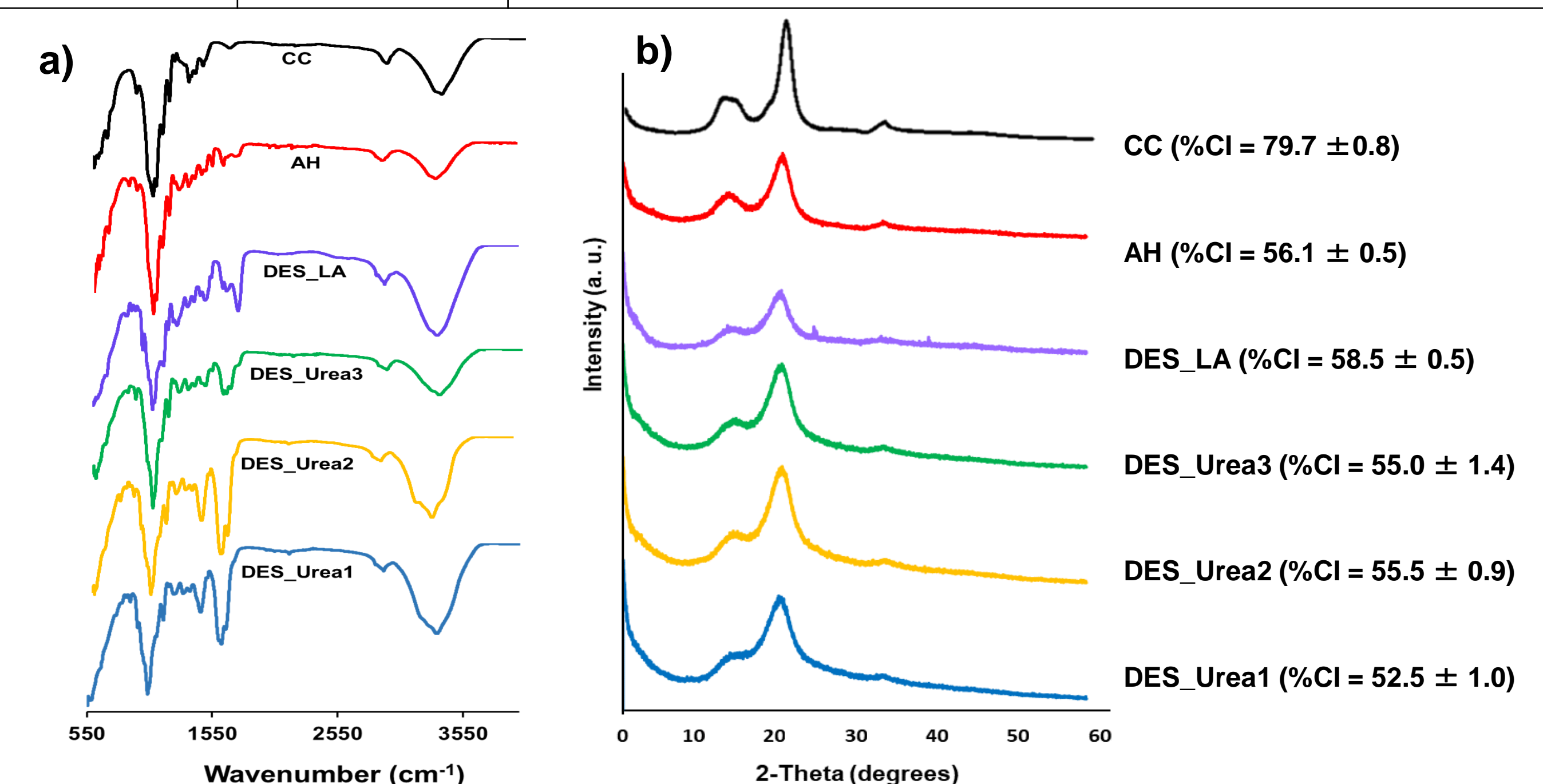


Figure 1. FT-IR spectra (a) and PXRD analysis (b) of the obtained solid fractions rich in cellulose, in comparison with the data obtained from a standard cellulose from Sigma-Aldrich (CC).

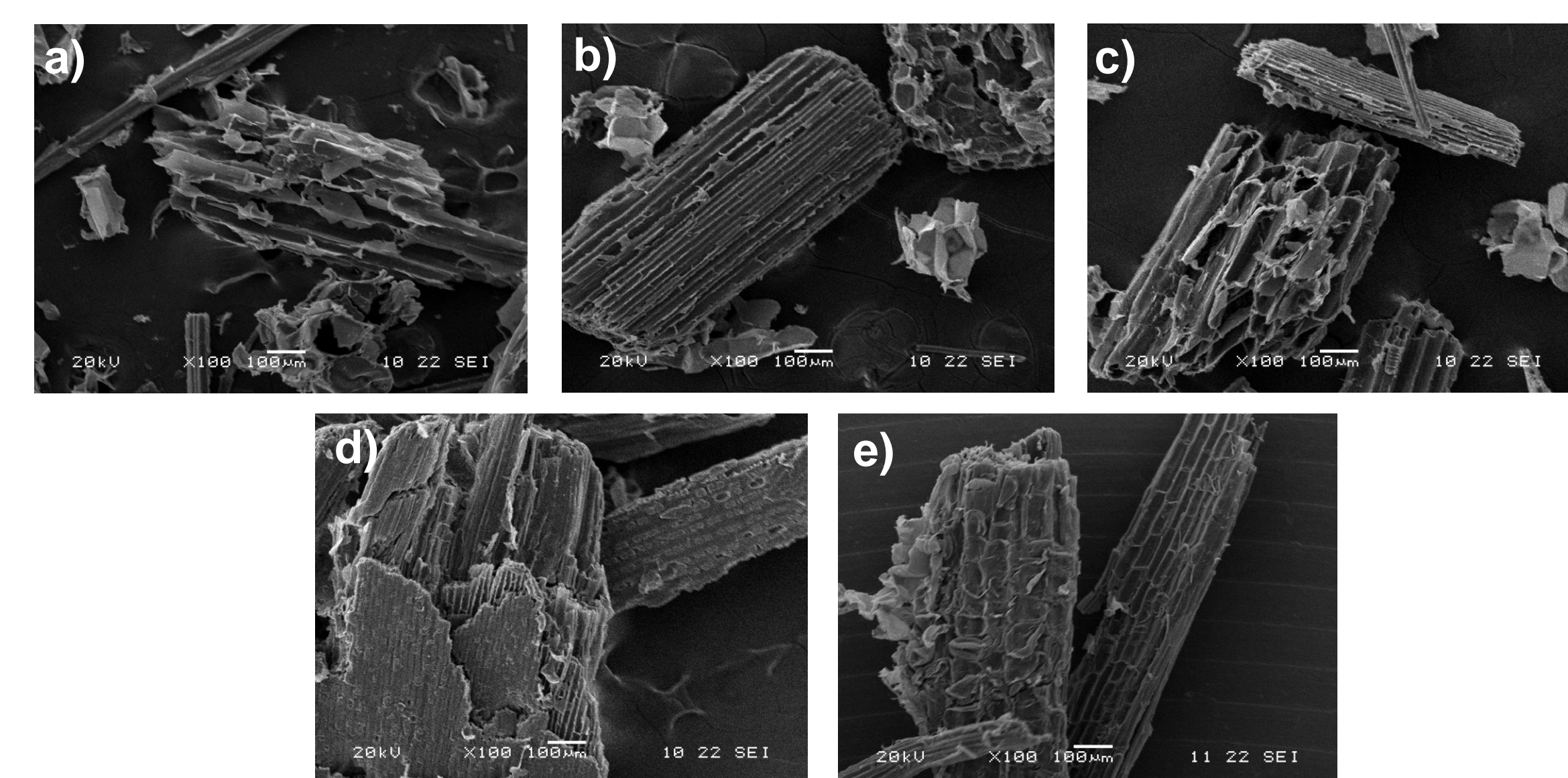


Figure 2. SEM images of the obtained solid fractions rich in cellulose: a) DES\_Urea1; b) DES\_Urea2; c) DES\_Urea3; d) DES\_LA and e) AH.

## Conclusions

Cellulose rich fractions were successfully obtained from SCB by sustainable methods. The autohydrolysis (AH) process was the most promising, producing a solid fraction richer in cellulose (i.e., 59.1%) and with less hemicellulose (i.e., 11.4%), when compared to the fractions obtained by DES (i.e., 27.8-34.7% cellulose and 17.7-25.9% hemicellulose). AH yielded a cellulose fraction with 56.2%, exhibiting a crystallinity degree of 56.1% (by PXRD) and typical cellulose functional groups (by FT-IR).

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## References

- Li YY, Wang B, Ma MG, Wang B (2018) Review of Recent Development on Preparation, Properties, and Applications of Cellulose-Based Functional Materials. *Int J Polym Sci*, 2018.
- Jonoobi M, Oladi R, Davoudpour Y, et al (2015) Different preparation methods and properties of nanostructured cellulose from various natural resources and residues: a review. *Cellulose* 22:935-969.
- Motaung TE, Mochane MJ (2017) Systematic review on recent studies on sugar cane bagasse and bagasse cellulose polymer composites. *J. Thermoplast. Compos. Mater*, 31(10):1416-1432.
- Radotić K, Mičić M (2016) Methods for Extraction and Purification of Lignin and Cellulose from Plant Tissues. Humana Press, New York, NY, pp 365-376.
- Chourasia VR, Pandey A, Pant KK, Henry RJ (2021) Improving enzymatic digestibility of sugarcane bagasse from different varieties of sugarcane using deep eutectic solvent pretreatment. *Bioresour Technol* 337:125480.
- Sasaki M, Adschiri T, Arai K (2003) Fractionation of sugarcane bagasse by hydrothermal treatment. *Bioresour Technol* 86:301-304.
- Sluiter A, Hames B, Ruiz R, et al (2008) Determination of Structural Carbohydrates and Lignin in Biomass: Laboratory Analytical Procedure (LAP) (Revised July 2011).