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Separation and Recovery of a Hemicellulose-Derived Sugar Produced from the Hydrolysis of Biomass by an Acidic Ionic Liquid

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Biomass processing with ionic liquids (ILs) has been one of the most topical research areas in recent years. However, separation and recovery of biomass products and ILs are currently a challenge. Recovery of produced monosaccharides from an IL postreaction solution and the possibility to reuse the IL are strongly required to guarantee the sustainability of biomass processing. The present study demonstrates a novel approach that aims at separating a biomass hemicellulose-derived product, namely, xylose, and 1-ethyl-3-methylimidazolium hydrogensulfate ([emim][HSO_4]). High polarity of a postreaction system composed of xylose, IL, and water is one of the major hindrances in the separation performance. A proposed solution is fine-tuning of the system polarity by the addition of moderately polar acetonitrile. To scrutinize the potential of xylose and IL separation, phase equilibria of a system constituted by [emim][HSO_4], water, and acetonitrile were studied. Additionally, preparative chromatography experiments with alumina as a stationary phase were performed to determine the conditions required for efficient separation of the sugar and the IL by selective adsorption of xylose on alumina in detriment of IL. The amount and treatment of the stationary phase, eluent polarity, and amount of loaded sample were also scrutinized in this study. Treatment of alumina was considered as a necessary step to achieve recovery yields of 90.8 and 98.1 wt% for the IL and xylose, respectively, as separate fractions.

Introduction

Carbohydrates are an ample class of organic compounds that are widespread in nature and responsible for structural and energy-source functions in biological systems.[1] One of the most important features of carbohydrates is their easy assimilation by microorganisms and facile conversion into a series of valuable compounds used in pharmaceutical and cosmetic applications,[2] food formulations,[3] biomedical devices,[4] nanomaterials,[5] and biotechnological processes.[6] The biotechnological processes -have been intensively explored in the frame of the biorefinery concept, and it consists of using microorganisms to convert monosaccharides, for example, glucose and xylose, into a myriad of chemicals (e.g., succinic acid and lactic acid), fuels (e.g., bioethanol and biobutanol), and value-added commodities (e.g., itaconic acid and polyhydroxyalkanoates). However, the price and availability of monosaccharides are key issues for the cost-effective accomplishment of this concept. Cereals are examples of biomass materials rich in fermentable carbohydrates, but food versus fuel concerns and land-use issues restrict their use for these purposes.[7] In this context, other feedstocks containing easily available carbohydrates have been sought. One of them is lignocellulosic biomass, which is composed up to 80 wt% polymeric carbohydrates, mostly in the form of cellulose (or starch) and hemicellulose[8] The physical, chemical, physicochemical, and/or biological treatments of lignocellulosic biomass allow the required monosaccharides to be obtained in upgradable forms.[9] Acid hydrolysis treatment stands as one of the principal options to achieve that. Nevertheless, this technology requires specialized reactors able to sustain high temperatures and pressures and that are corrosion resistant. Additionally, acid neutralization negatively affects the environmental aspects of biomass processing. Therefore, greener and more sustainable technologies, for example, the use of ionic liquids (ILs), have emerged.[9,10] Ionic liquids are organic salts, and as a result of numerous potential combinations of cations and anions, ILs are called “designer solvents”.[11] This allows the construction of ILs with task-specific and unique properties, such as thermal stability, nonflammability, and a wide range of polarities, acidities, and basicities among others.[12] Additionally, ILs possess great solvent power towards a large variety of chemicals, including polymers.[13] All of these features have driven the full exploitation of ILs in diverse fields, including biomass processing.[14] In this context, one of the most recent trends is biomass intensification, which involves integrating biomass dissolution with hydrolysis by the use of acidic ILs.[15] However, similar to other applications of ILs, two main challenges remain, that is, separa-

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