ADSORPTION SEPARATION PROCESSES FOR IONIC LIQUID CATALYTIC PROCESSES

CROSS REFERENCE TO RELATED APPLICATION

This application claims the benefit of U.S. Provisional Patent Application No. 61/258,347, filed Nov. 5, 2009, which is hereby incorporated by reference in its entirety.

ACKNOWLEDGMENT OF GOVERNMENT SUPPORT

This invention was made with Government support under Contract DE-AC06-76RLO1830 awarded by the U.S. Department of Energy. The Government has certain rights in the invention.

FIELD

Disclosed are methods for separation of products, byproducts and ionic liquids from ionic liquid catalysis process reaction mixtures, and recycling of ionic liquids, particularly separation of mixtures from ionic liquid catalysis processes for the conversion of biomass, cellulose, and sugars into chemical intermediates, such as hydroxymethylfurfural.

BACKGROUND

Ionic liquids, due to their unique attributes, are a new generation of high performance solvents useful for catalytic and separation applications. Ionic liquid-based reactions are considered to be "green" compared to conventional solvents because they possess superior properties; they have relatively low vapor pressures, tend to be non-flammable or essentially non-combustible below their decomposition temperature, have excellent thermal stability with a wide range of tunable liquid properties, and are superior solvents for a diverse array of compounds. The ionic liquids' properties result in liquid carriers that provide operational flexibility and minimize the environmental footprint of a process.

For catalysis systems in ionic liquids, the ionic liquid can act as a solvent (liquid carrier) and/or as a catalyst to stabilize reaction intermediates. However, a major barrier hindering industrial use of ionic liquids is the high costs; the ionic liquid cost is often greater than the reaction product mixture and desired product cost itself. For a practical process, the ionic liquid should be recovered from the reaction mixture for re-use at a very high recovery rate (greater than 80% or 90% or most likely greater than 99 or 99.9%).

The separation technologies currently in use in such ionic liquid catalysis process systems are distillation, vacuum distillation or extraction. Those techniques provide low separation efficiency for many desired products, are not readily scalable to a commercial level, have an unacceptable effect on the environment, and/or are not safe for workers. In addition, certain of the currently used techniques require raising reaction mixtures to boiling temperatures and/or cooling the same-energy intensive activities harmful to the environment and costly on a commercial scale. In distillation processes the required heating levels of the reaction product mixture necessary to cause separation also result in many side reactions that are detrimental to recovery of product, feed, catalyst, and/or the ionic liquid. Extraction processes use particular organic solvents and apparatus that cause the process not only to be more complicated on a commercial scale (as opposed to lab bench processes) but also produce undesirable solvent waste.

Hydroxymethylfurfural (HMF) is a key intermediate chemical and is a flexible platform for producing chemicals and fuels that can substitute for today's petroleum-derived reaction product mixtures. Recent developments have been made in processes for the production of HMF on a commercial scale and at costs that allow petroleum reaction product mixture substitution in the production of major chemicals from biomass and biobased fuels. Scientists at Pacific Northwest National Laboratories have developed a number of inventions demonstrating catalytic conversion of sugars to HMF at high selectivities and conversions using a soluble catalyst in ionic liquid solvents. Such processes require a separation of unreacted sugars, HMF and ionic liquids.

Efficient, cost effective and environmentally sound separation processes for ionic liquid catalysis process systems are needed to recover important reaction components such as the ionic liquids as well as the reaction product and byproducts and allow for the recycling of the ionic liquid on a commercially economically and environmentally acceptable scale.

SUMMARY

Separation of product molecules from ionic liquid reaction mixtures has been a challenge for processes using ionic liquids as solvents (carriers) and/or catalysts. This is particularly true for HMF; due to the high boiling points of many of the useful ionic liquids and the instability of HMF and sugar at high temperatures, distillation separation is not a practical option in HMF generating processes. Separation of HMF by extraction encounters a lack of highly selective solvent and the formation of emulsion between the ionic liquids and extraction solvents. The existing conventional separation processes are not economically and/or environmentally acceptable on a commercial production scale. In addition, such conventional methods do not allow for the cost-effective and/or environmentally friendly recovery of the ionic liquid on a commercial-production scale. The presently disclosed adsorption separation processes utilize researched and developed adsorbent materials in a fixed structure. The ionic liquid reaction mixtures flow through a bed of particularly chosen adsorbent materials and the desired product molecule, such as HMF, is selectively retained by the adsorbent material while the costly ionic liquid solvent is eluted for re-use. The saturated adsorbent material is then regenerated (or desorbed) for recovery of the HMF. The presently disclosed processes allow such separations to be performed economically and in an environmentally sound manner, on a commercial production scale.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a process flow diagram for a virtually completely "green" continuous flow, ionic liquid-based catalytic process for production of HMF from low-cost sugar reaction product mixtures including embodiments of the presently disclosed adsorption processes.

FIG. **2** illustrates certain apparatus useful for practicing the disclosed separation and regeneration processes.

FIG. **3** illustrates certain apparatus useful for practicing the disclosed regeneration processes for regenerating a saturated adsorbent material bed.

FIG. **4** illustrates apparatus for an embodiment of the disclosed separation process that is integrated directly with an HMF ionic liquid catalytic production process.