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Multiphase Hydrogenation of D-Glucosamine Hydrochloride, N-Acetyl-D-glucosamine, D-Glucose, and D-Maltose over Ru/C with Integrated Catalyst Recovery

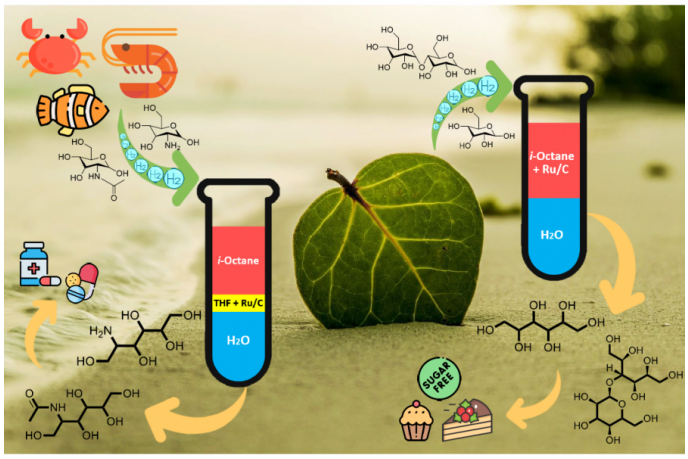
AUTHORS

Daniele POLIDORO / CA' FOSCARI UNIVERSITY OF VENICE, VIA TORINO 155, VENEZIA
Corresponding author : MAURIZIO SELVA / selva@unive.it

PURPOSE OF THE ABSTRACT

A multiphase system (MP) comprised of two immiscible liquids, water and isooctane, and commercial 5% Ru/C as a catalyst, both with and without an additional organic liquid (OL: tetrahydrofuran (THF), 2-methyl-THF, methyl isobutyl carbinol, and cyclopentyl methyl ether) was designed and investigated for the chemoselective catalytic hydrogenation of four model examples of bio-based sugars and amino/amido-sugars. At 110 °C and 40 bar of H2, D-glucosamine hydrochloride and N-acetyl-D-glucosamine were converted selectively into their corresponding hydrogenated derivatives, 2-amino-D-sorbitol and 2-acetamide-D-sorbitol, respectively, isolated in >99% yields. Both the reagents and the products were converted and formed in the aqueous phase, respectively; while, by tuning the relative proportions of water, isooctane, and the third added liquid (particularly tetrahydrofuran), the catalyst (Ru/C) was perfectly segregated in the organic layer, where it could be recycled and reused up to 9 times without any loss of activity and selectivity, in a semi-continuous mode. Under such conditions, the reaction was implemented on a gram scale with a productivity up to 0.89 mmol 2-amino-D-sorbitol/(gcat h). The same hydrogenation efficiency and reagent/products/catalyst separation were achieved during the multiphase reactions of D-glucose and D-maltose. In this case, however, results were independent from the multiphase composition: at 120 °C and 20-40 bar of H2, using either H2O/isooctane or H2O/isooctane/OL systems, a quantitative conversion of D-glucose and D-maltose was reached with a selectivity up to 78% and >99% towards sorbitol and maltitol, respectively. Ru/C was perfectly separated out of the aqueous phase in both multiphase mixtures, with a negligible metal leaching, below 0.01 wt%.
The multiphase approach for all the tested substrates proved not only an original and robust protocol to improve the products isolation and catalyst recycle, but also effective in preventing metal contamination in the synthesis of final derivatives.

FIGURES



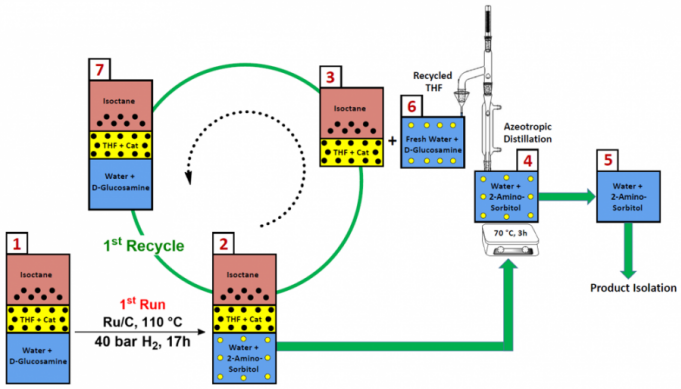


FIGURE 1

Work Overview

Overview of all investigated reactions

FIGURE 2

Semicontinuous catalytic hydrogenation of D-Glucosamine under MP-conditions

Semicontinuous catalytic hydrogenation of D-Glucosamine under MP-conditions with integrated recycling of the catalyst and recovery of THF.

KEYWORDS

Multi Phase System | Green Chemistry | Clean Reaction | Biomass

BIBLIOGRAPHY

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