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Optimization of Microwave-Assisted synthesis of 5-hydroxymethyl-2-furfural

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Introduction

Heterocyclic furan-based compounds are interesting bio-renewable materials used as intermediate for pharmaceuticals, biodegradable resins, biofuels and fine chemicals syntheses. Additionally, HMF also functions as an anti-sickling agent for intermolecular sickle hemoglobin without inhibition by plasma and tissue proteins or other undesirable sequences [1].

Experimental

In this work, the optimization procedure of HMF (Figure 1.) microwave-assisted D-fructose dehydration in hydrochloric acid or ionic liquid (1-butyl-3-methylimidazolinium bromide) (IL) was performed. HMF was further subjected to oxidation and reduction processes to obtain 2,5-furandicarboxylic acid and furan-2,5-diyldimethanol, respectively, useful reactants in a biorenewable based polyester resins synthesis (Figure 1.).

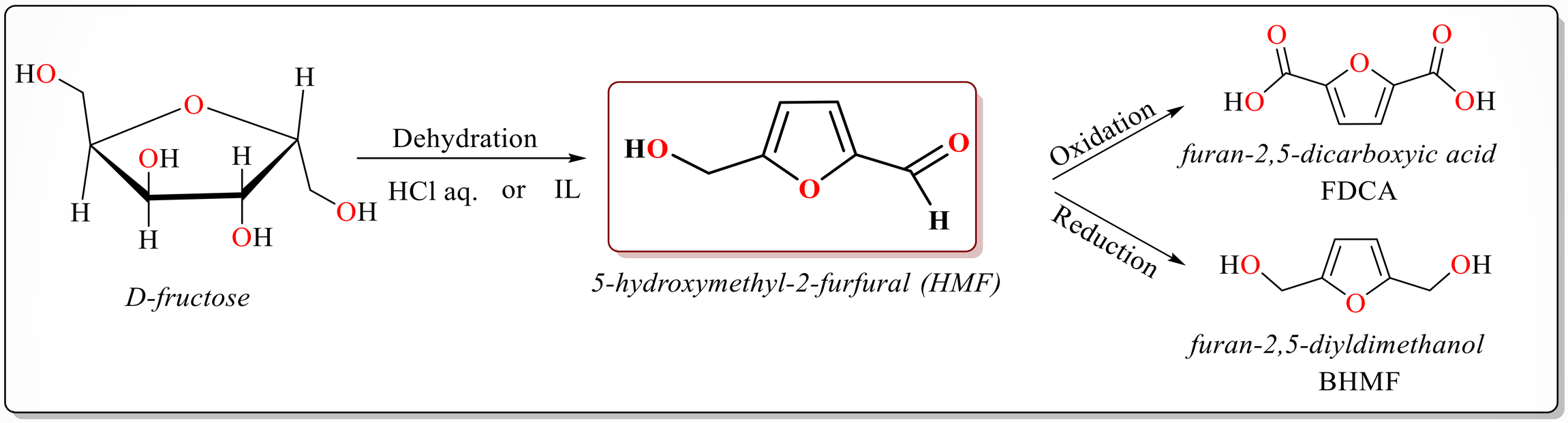

Figure 1 Optimization procedure of HMF synthesis and its possible transformation to useful products

Results and disscusion

The structure and purity of HMF were confirmed according to results from elemental analysis, ATR-FTIR and NMR spectroscopy. In order to obtain the highest yield and purity of the product optimization of HMF synthesis was performed with respect to pH, temperature, irradiation power and time of reaction.

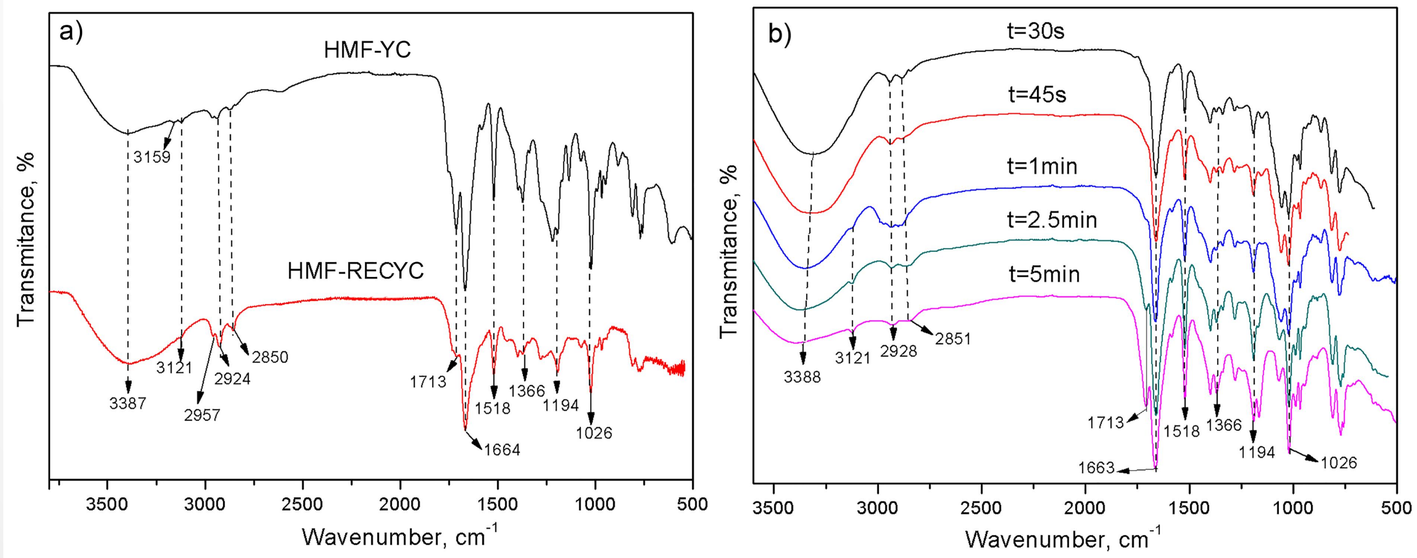

Figure 2 a) FTIR spectra of HMF obtained from YC and recycling of YC b) and reaction time of HMF

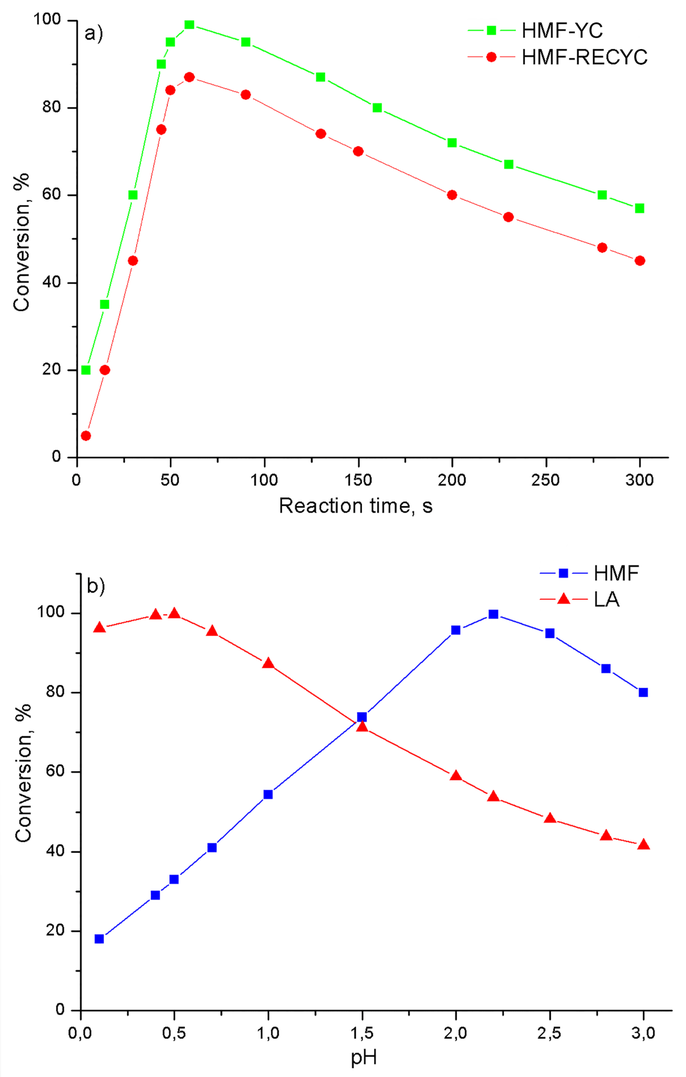

Figure 3 a) Results of optimization of HMF in relation to reaction time, and b) pH

Conclusions

Results of the HMF optimization with respect to reaction time and recycling IL show that the highest yield and purity were obtained for 50 s, and a further increase of reaction time influence the increase of the levulinic acid production (Fig 2. and 3a). On the other hand, lower pH is favorable for HMF production (~0.5), while higher (~2.3) leads to a higher yield of levulinic acid (Fig. 3b). The usefulness of the applied optimization method was confirmed by HMF oxidation (O2/V2O5) and reduction (NaBH4) to produce 2,5-FDCA and BHMF as useful reactants in bio-renewable polyester synthesis.

Reference

[1] Maggel Deetlefs and Kenneth R. Seddon, Green Chemistry, 2003, 5, 181-186

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