Increasing the selectivity of HMF formation in aqueous solution using *in situ* adsorption

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Introduction

The selectivity of fructose dehydration to HMF in aqueous solutions is low because HMF hydrolyses to levulinic acid and formic acid. In order to reduce the hydrolytic decomposition of HMF its removal from the aqueous solution by an *in situ* adsorption approach was tested to increase the overall selectivity of HMF formation. Further on, the use of sucrose as cheap fructose source was tested. After the instanteous hydrolysis of sucrose fructose reacts to HMF whereas glucose remains unchanged.

Results

➔ Screening for adsorbents

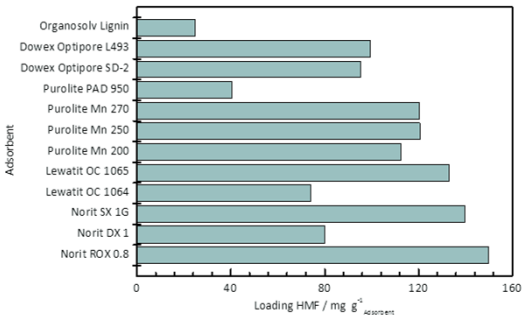

Fig. 1: *HMF adsorption in water at different adsorbents at room temperature, 100 mmol·L^{-1} HMF, 2 g adsorber.*

➔ Adsorption isotherms

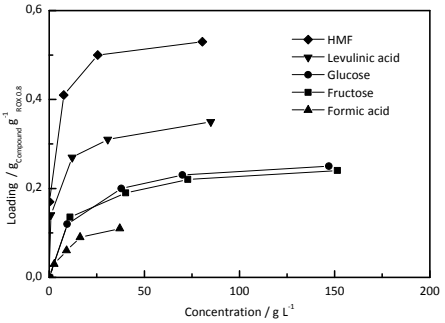

Fig. 2: *Adsorption isotherms of HMF, levulinic acid, formic acid, glucose and fructose at room temperature for activated carbon type ROX, 1M HCl as solvent, 2 g ROX 100-1000 mmol·L^{-1} substances.*

➔ Desorption

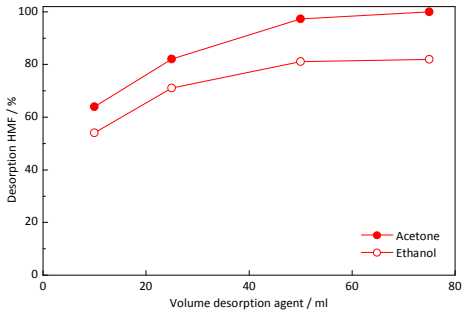

Fig. 3: *HMF Desorption in dependence of the acetone or ethanol volume at room temperature, HMF loading ≈ 0,4 g/g, 0-75 mL Aceton/Ethanol, 2 h.*

- Activated carbon type ROX has the highest HMF adsorption capacity
- high HMF loading: 0.5 g_{HMF} / g_{ROX}
- no pH dependency of the adsorption between pH 0-7
- no temperature dependency of the adsorption at T = 60 °C and 80 °C
- complete HMF desorption with acetone as solvent (boiling point = 56°C)

➔ HMF formation with *in situ* adsorption

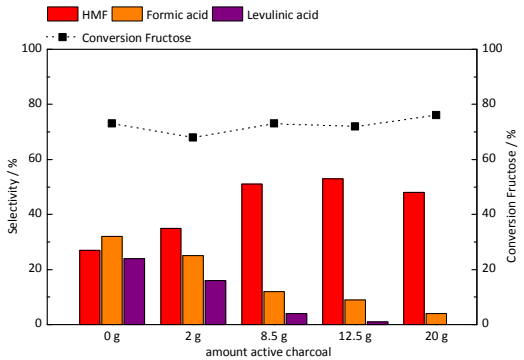

Fig. 4: *HMF selectivity in aqueous solution in dependence of the amount of activated carbon at equal conversions of the fructose moiety of the sucrose, 662 mmol·L^{-1} sucrose, 20 wt.-% HCl (50 mL), 0-20 g ROX, 60 °C, 2 h.*

- Increase of HMF selectivity from 27 % (without adsorber) to > 50 % (with adsorber)
- No significant change in HMF selectivity at batch or continuous reaction conditions
- Approx. 20 % of the glucose moiety from the sucrose react as well, probably unwanted by-products such as humins are formed, which were not further specified

Summary

The HMF formation selectivity can be doubled by *in situ* adsorption at the activated carbon ROX. A complete recovery of the adsorbed HMF from the activated carbon is achieved by acetone as solvent (low boiling point).