PRA INOVAR TEM QUE PESQUISAR.

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BIOMASS VALORIZATION: CONVERSION OF FRUCTOSE INTO 5-HYDROXYMETHYLFURFURAL BY HETEROGENEOUS CATALYSIS

LBIOP Laboratório de Biotecnologia de Produtos Naturais e Sintéticos

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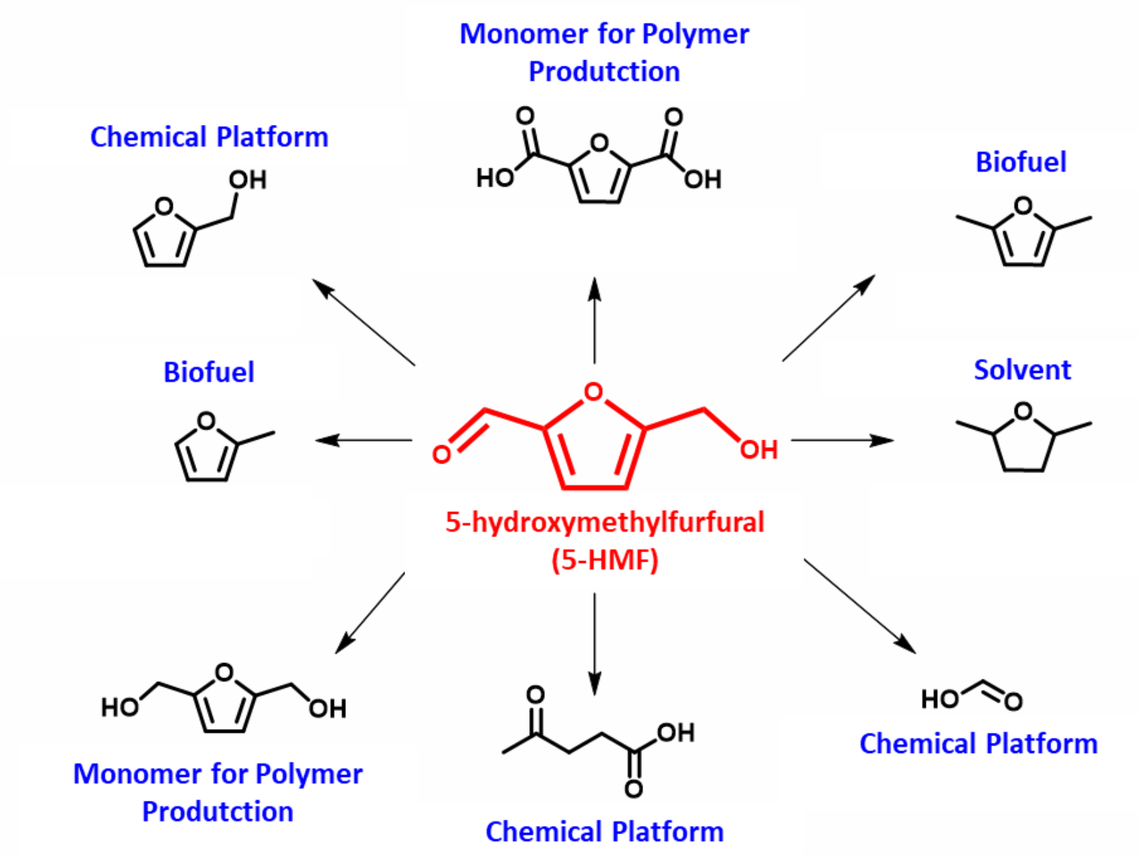
Introduction

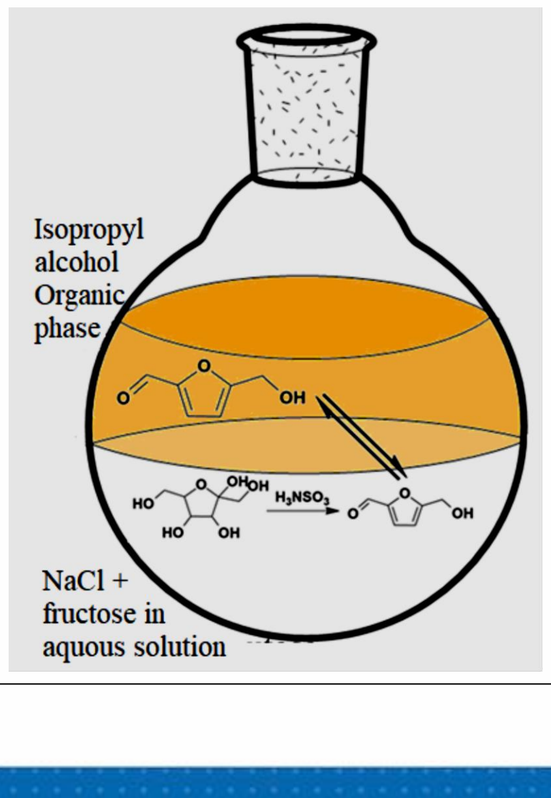
In recent years, catalytic production methods of converting biomass into furan derivatives have been improved significantly. One of these furan derivates is the 5-hydroxymethylfurfural (5-HMF), that can be obtained from carbohydrate dehydration, mainly C5 and C6.[1,2]



The aim of this work was to evaluate how reactional conditions, such as temperature, reaction time and catalyst amount influence the conversion and selectivity of fructose into 5-HMF using microwave radiation.

Experimental

In a 30 mL vial were added fructose (90 mg, 0,5 mmol), saturated solution of sodium chloride (3 mL), 2-propanol (6 mL) and sulfamic acid (10 and 20 mol%). The vial was sealed and submitted to microwave irradiation (Anton Paar Monowave 300) at temperatures (150 and 180 °C) and times (10 and 20 minutes). Temperature, time and catalyst amount were stablished by a Design of Experiment (DOE).



After the complete of the reaction, the solution was left to chill at room temperature. The aqueous and organic phases were separated and analyzed by high efficiency liquid chromatography (HPLC).

Results and Discussion

All measurements of the concentrations was made by calibrations curve of 5-HMF and fructose. After, were calculated the conversion and selectivity.

Table 1: Quantification of HPLC data for fructose and 5-HMF in experiments outlined by design of experiment (DOE).

Experiment	t (min)	T (°C)	Catalyst (mol%)	[ ]Fructose (mmol)	[ ] 5-HMF (mmol)
E1	10	150	10	0.31±0.01	0.21±0.01
E2	10	150	20	0.23±0.02	0.26±0.015
E3	10	180	10	0.055±0.015	0.56±0.005
E4	10	180	20	0.015±0.005	0.39±0.035
E5	20	150	10	0.165±0.045	0.34±0.01
E6	20	150	20	0.21±0.09	0.29±0.07
E7	20	180	10	0.02±0.00	0.38±0.02
E8	20	180	20	0.015±0.005	0.38±0.015

From the results obtained from the DOE, and summarized in Table 2, the temperature is the variable that most influences the conversion and selectivity. Reaction time of 10 minutes, temperature of 180 °C and catalyst amount of 20mol% were found as the best reaction conditions, with 96,84% of conversion of fructose and 76,72% of selectivity for 5-HMF formation (Table 2, E4).

Table 2: Conversion and selectivity resulting from the reaction of fructose into 5-HMF.

Experiment	t (min)	T (°C)	Catalyst (mol%)	Conversion (%)	Selectivity (%)
E1	10	150	10	37.22±2.23	42.04±2.50
E2	10	150	20	53.08±3.94	45.96±3.03
E3	10	180	10	89.32±2.58	71.33±1.53
E4	10	180	20	96.47±0.59	76.72±7.51
E5	20	150	10	64.77±7.25	68.54±1.73
E6	20	150	20	57.96±18.45	59.57±14.91
E7	20	180	10	95.75±0.75	75.81±3.53
E8	20	180	20	96.84±1.63	74.94±2.47

Conclusion

In the present work, it was possible to successfully evaluate the conversion of fructose into 5-HMF with high selectivity using sulfamic acid as a catalyst.

The methodology developed in this work proved to be simple and safe following the 12 principles of green chemistry, as well as the 17 United Nations sustainable development goals.

Bibliographic references

[1] Galaverna, R.; Pastre, J. C. *Rev. Virtual Quim.* **2017**, *9*, 248-273.

[2] Zhang, Z., Du, B., Zhang, L-J., Da, Y-X., Quan, Z-J., Yang, L-J., Wang, X-C. *RSC Adv.* **2013**, *3*, 9201-9205.

Anknoledgements

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