The effect of mechanochemistry on the preparation of heterogeneous catalysts: Reduction of furfural to obtain furfuryl alcohol

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Introduction

In recent decades, the production routes of chemicals are being influenced by the principles of Green Chemistry. This can improve yields and reduce the environmental impact, as well as provide alternative feedstock to fossil resources as raw materials for these chemical processes, or even modify the production chain.

The search for green processes in a circular economy framework opens numerous lines of research to valorize biomass wastes to obtain valuable products. In this context, mechanochemistry is receiving increasing attention for the synthesis of many chemicals in the solid state, being considered by IUPAC as one of the 10 methodologies to change the world.

Hemicellulose and cellulose contained in lignocellulosic biomass can be hydrolyzed into their corresponding monomeric carbohydrates, mainly C5 and C6 sugars, respectively. These, in turn, can be dehydrated to obtain furfural and 5-hydroxymethylfurfural. Furfural is a platform molecule from which furfuryl alcohol is obtained by hydrogenation, being an intermediate for the synthesis of a large spectrum of chemicals of high industrial interest, such as 1,5-pentanediol or 2-methyltetrahydrofuran.

In the present work, catalytic transfer hydrogenation (CTH) in the presence of 2-propanol, as a hydrogen donor, has been used as an alternative to H_2.[1,2,3]

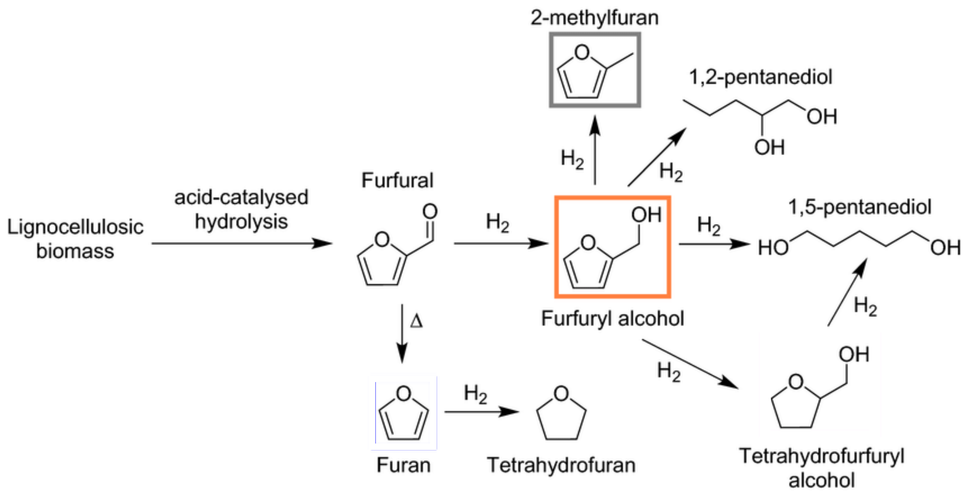

Figure 1. Chemicals derived from the hydrogenation of furfuryl alcohol
Reference: MacIntosh and Beaumont, 2020

Results

$Mg(OH)_2$ was mechanochemically treated in order to decrease particle size and improve its catalytic properties at different times, in the DYNO®-MILL RESEARCH LAB system. Then, it was calcined at 450ºC to obtain MgO, which is the catalyst.

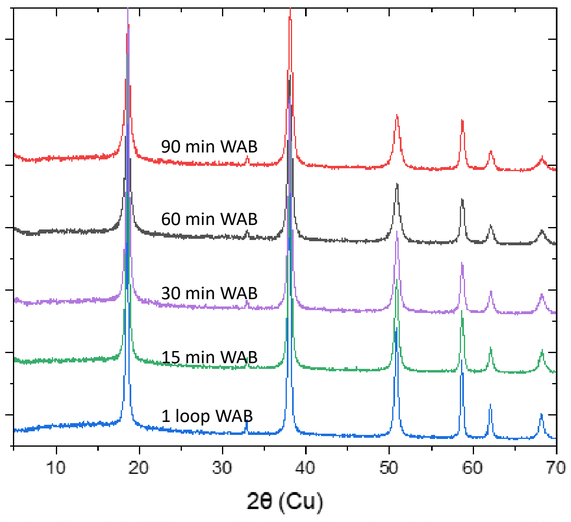

Figure 2. XRD of MgO obtained from $Mg(OH)_2$ treated in the mechanochemical system at different times (10 wt% loading).

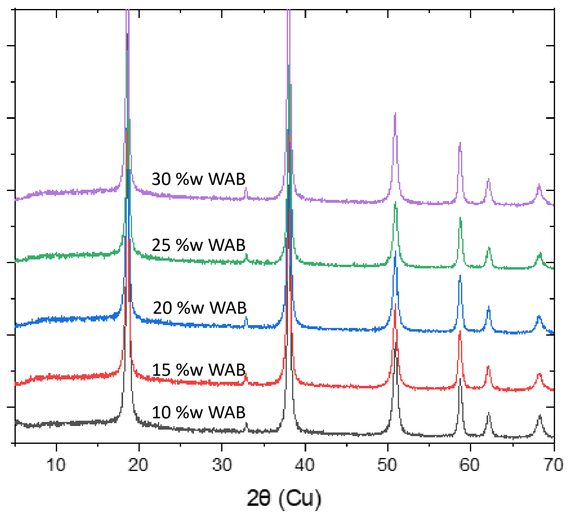

Figure 3. The effect of the $Mg(OH)_2$ loading treated in the mechanochemical system for 15 min on the XRD pattern of the resulting MgO.

Catalyst test:

1) Influence of the mechanochemical treatment for $Mg(OH)_2$ on the catalytic performance of MgO

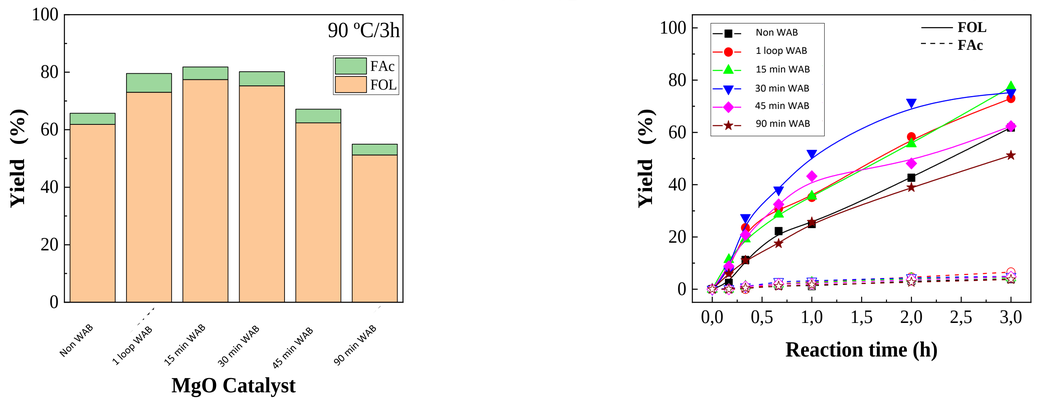

Figure 4. Reduction of furfural to furfuryl alcohol by catalytic transfer hydrogenation (CTH).
Hydrothermal = Batch
Temperature: 90ºC ; MgO obtained at different residence times; 2 ; propanol/furfural molar ratio: 50 ; furfural/catalyst weight ratio: 1

2) Catalytic performance after different $Mg(OH)_2$ loadings during the mechanochemistry treatment

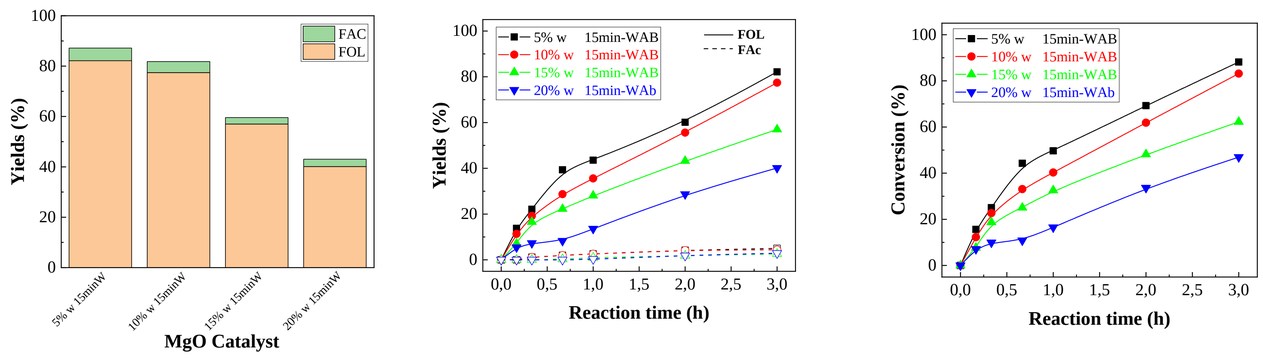

Figure 5. Reaction of furfural to furfuryl alcohol with different mechanochemically treated catalyst loadings.
Hydrothermal = Batch
Temperature: 90ºC ; MgO obtained at different loadings treated in the RL (15 min) c-MgO ; 2 ; propanol/furfural molar ratio: 50 ; furfural/catalyst weight ratio: 1

3) Effect of the reaction temperature on the catalytic performance:

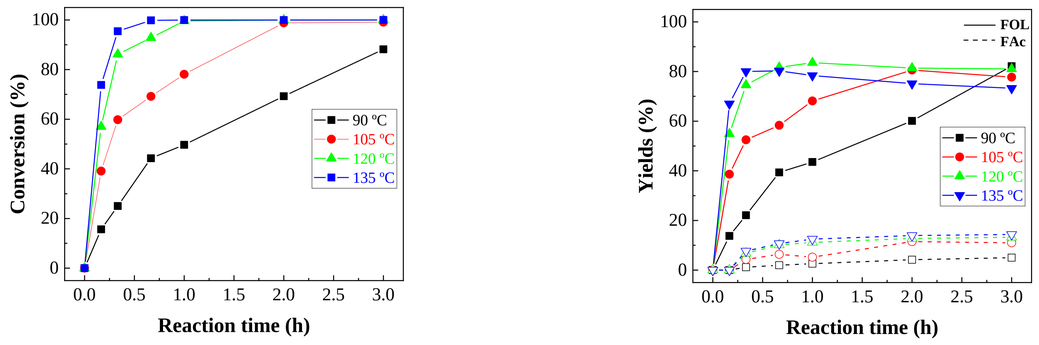

Figure 6. Conversion and yield of furfuryl alcohol at different reaction temperatures.
Hydrothermal = Batch
Temperature: 90ºC – 135 ºC ; Catalyst = c-MgO 15 min WAB; 2 ; propanol/furfural molar ratio: 50 ; furfural/catalyst weight ratio: 1

4) Reusing study of the catalyst:

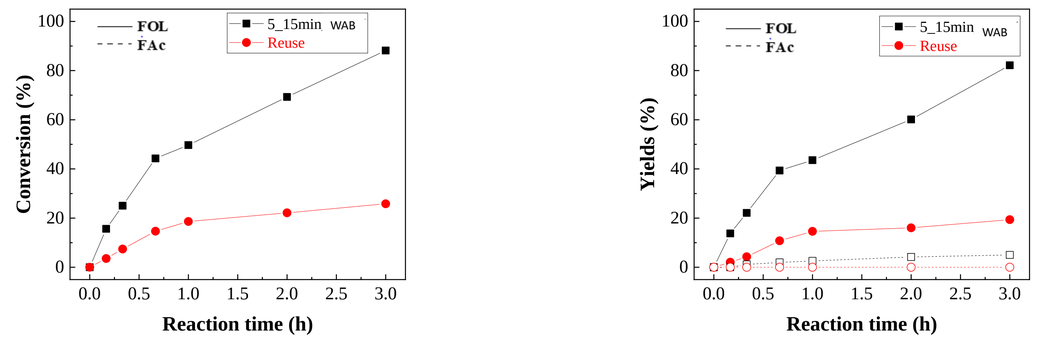

Figure 6. Reuse of MgO in the furfural hydrogenation reaction.
Hydrothermal = Batch
Temperature: 90ºC ; Catalyst = MgO 15 min WAB; 2 ; propanol/furfural molar ratio: 50 ; furfural/catalyst weight ratio: 1

Conclusion

The production of furfuryl alcohol has been successfully carried out from furfural by catalytic transfer hydrogenation, with high selectivity and FOL yield. Furthermore, the potential of the DYNO®-MILL RESEARCH LAB equipment for improving the catalytic properties of catalysts is demonstrated.

Bibliography

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