Supporting Information

Citral to Menthol Transformations in a Continuous Reactor over Ni/Mesoporous Aluminosilicate Extrudates Containing Sepiolite Clay Binder

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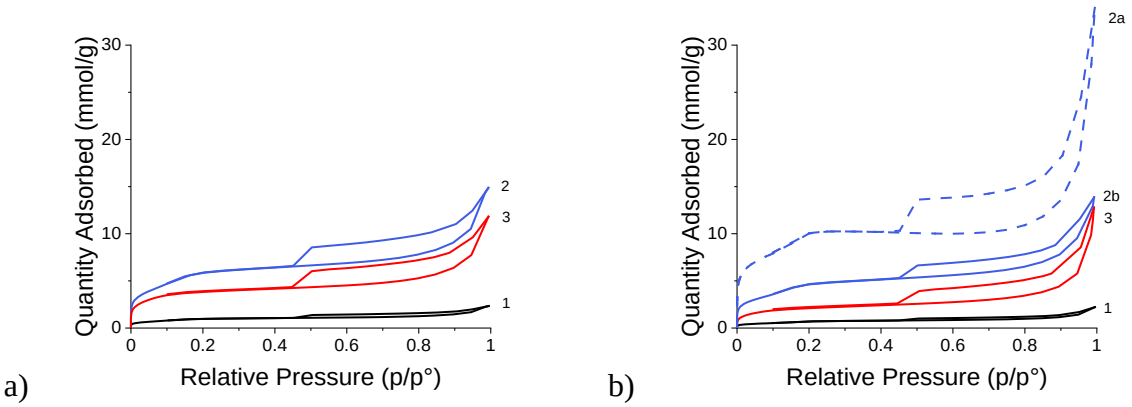
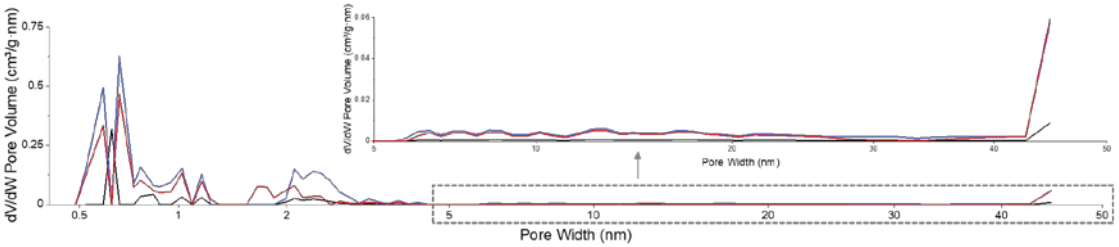
1. Characterization by N_2 physisorption

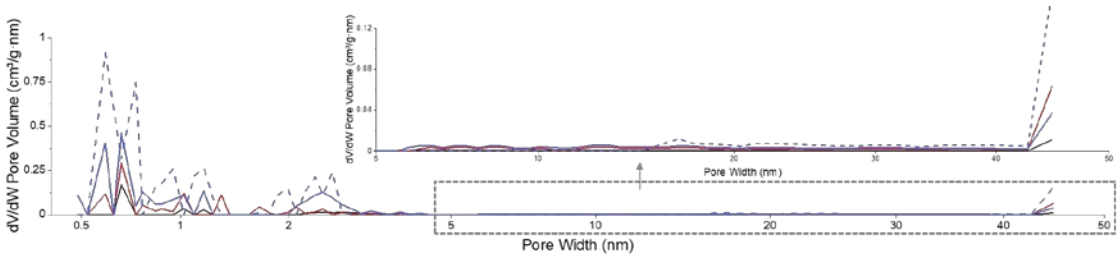


a) b)

Figure S1. Isotherms of a) powder catalysts: 1 - Ni/MAS fresh non-reduced powder catalyst (black), 2 - Ni/MAS fresh reduced powder catalyst (blue), 3 - Ni/MAS spent powder catalyst (red); b) extrudates: 1 - Ni/(MAS+sepiolite) fresh non-reduced extrudates (black), 2a - Ni/(MAS+sepiolite) fresh reduced crushed extrudates (blue, dash line), 2b - Ni/(MAS+sepiolite) fresh reduced extrudates (blue, solid line), 3 - Ni/(MAS+sepiolite) spent extrudates (red).



a)



b)

Figure S2. Pore size distribution of catalysts a) powder catalysts: 1 - Ni/MAS fresh non-reduced powder catalyst (black), 2 - Ni/MAS fresh reduced powder catalyst (blue), 3 - Ni/MAS spent powder catalyst (red); b) extrudates: 1 - Ni/(MAS+sepiolite) fresh non-reduced extrudates (black), 2a - Ni/(MAS+sepiolite) fresh reduced crushed extrudates (blue, dash line), 2b - Ni/(MAS+sepiolite) fresh reduced extrudates (blue, solid line), 3 - Ni/(MAS+sepiolite) spent extrudates (red).

2. XRD data of powder Ni/MAS catalysts

Powder XRD characterization of powdered catalysts was e performed using PANalytical Empyrean diffractometer with five axis goniometer. The incident beam optics consisted of Bragg-Brentano HD x-ray mirror, fixed 1/4◦ divergence slit, 10 mm mask, 0.04 rad soller slit and 1◦ antiscatter slit. The diffracted beam optics consisted of 7.5 mm divergence slit, 0.04 rad soller slit and PIXcel detector array. The used X-ray tube was Empyrean Cu LFF. The X-ray radiation was filtered to include only Cu Kα1 and Cu Kα2 components. The results were analyzed with MAUD (Material Analysis Using Diffraction) analysis program [1]. Instrumental broadening was evaluated with Si standard sample. The measured samples are as listed in Table S1. The results were obtained with $\theta - 2\theta$ scan range from 5◦ to 120◦.

Table S1. The description of Ni/MAS powder catalysts

#	Sample ID	Description
1	5%-Ni/H-MCM-41 PF	Powder fresh non-reduced
2	5%-Ni/H-MCM-41 PFR	Powder fresh reduced
3	5%-Ni/H-MCM-41 PS	Powder spent

The results for 5%-Ni H-MCM-41 PF, 5%-Ni H-MCM-41 PFR and 5%-Ni H-MCM-41 PS are presented in Figure S3. Figure S3 includes also the standardized diffraction patterns for Ni [2] and NiO [3]. The diffraction patterns presented in Figure S3 show a clear contribution from Ni in 5%-Ni H-MCM-41 PFR and 5%-Ni H-MCM-41 PS and NiO in 5%-Ni H-MCM-41 PF and 5%-Ni H-MCM-41 PFR. The peaks close 44.5^o, 51.8^o, 76.4^o and 93.0^o are assigned to Ni phase and the peaks close to 37.2^o, 43.3^o, 62.8^o, 75.4^o and 79.4^o to NiO. All the samples show also a broad diffraction peak close 20 ^{o}.

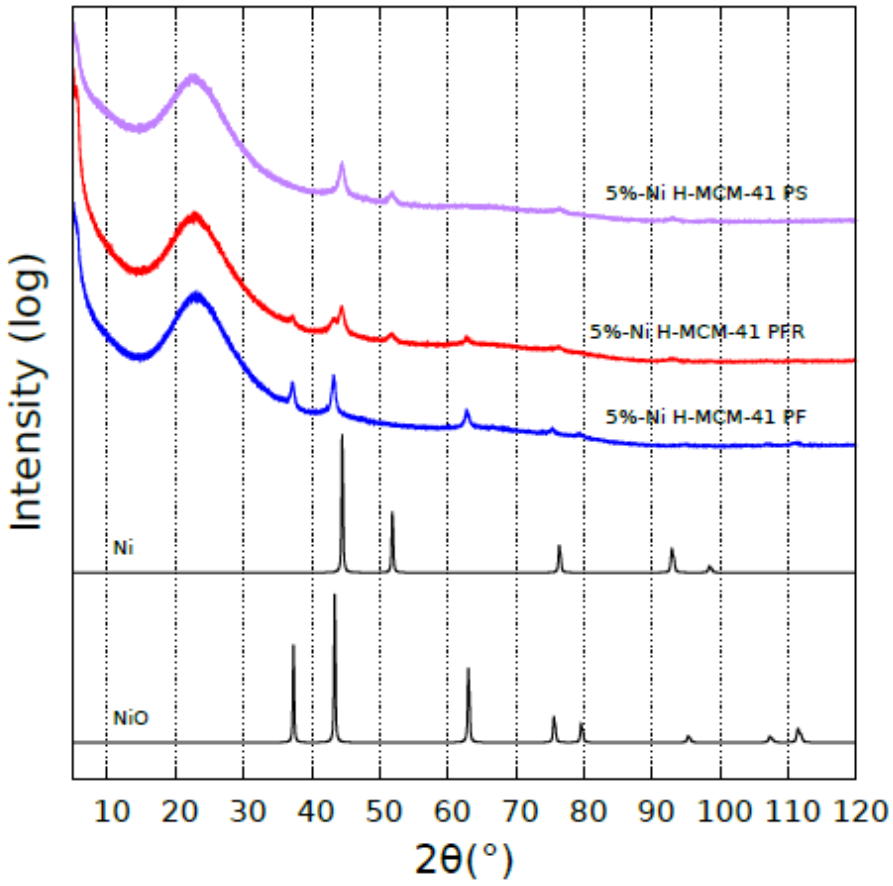
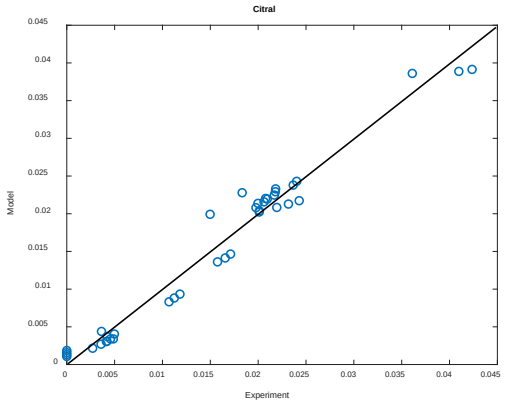
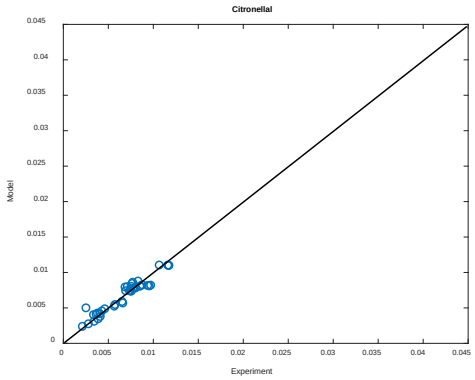


Figure S3. Combined results for 5%-Ni H-MCM-41 PF, 5%-Ni H-MCM-41 PFR and 5%-Ni H-MCM-41 PS along with standardized diffraction data for Ni and NiO.

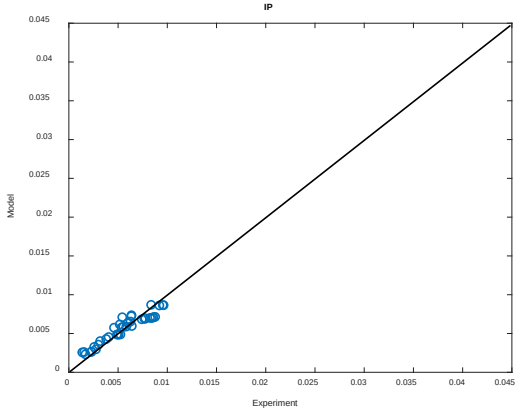
3. Kinetic modeling in continuous regime



a)



b)



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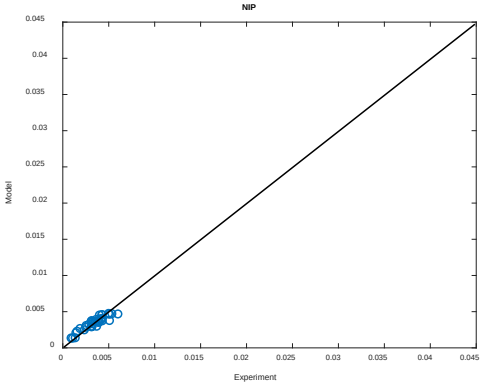
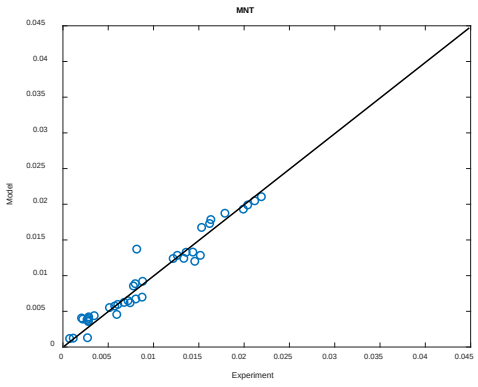
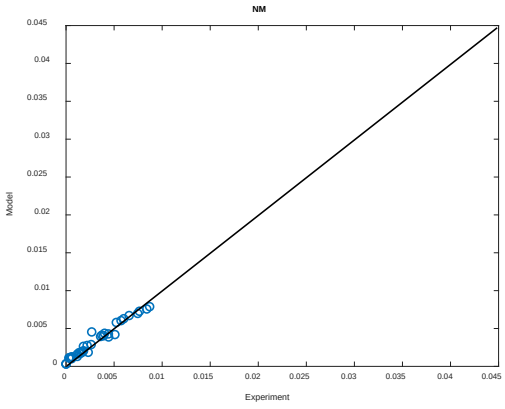


d)



e)



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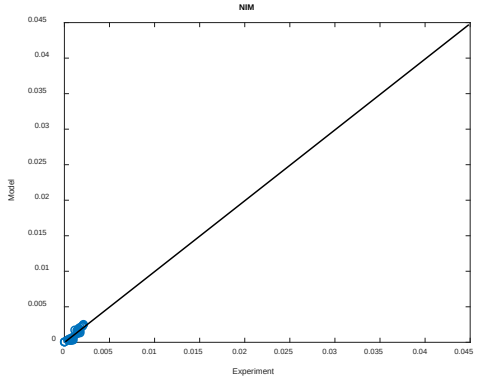

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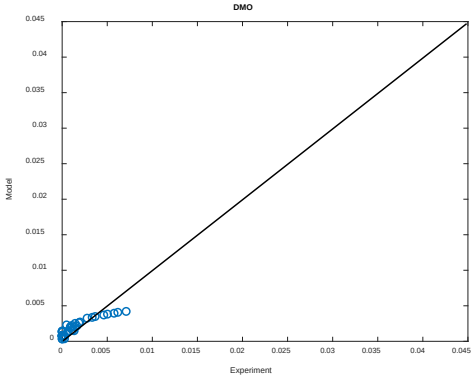

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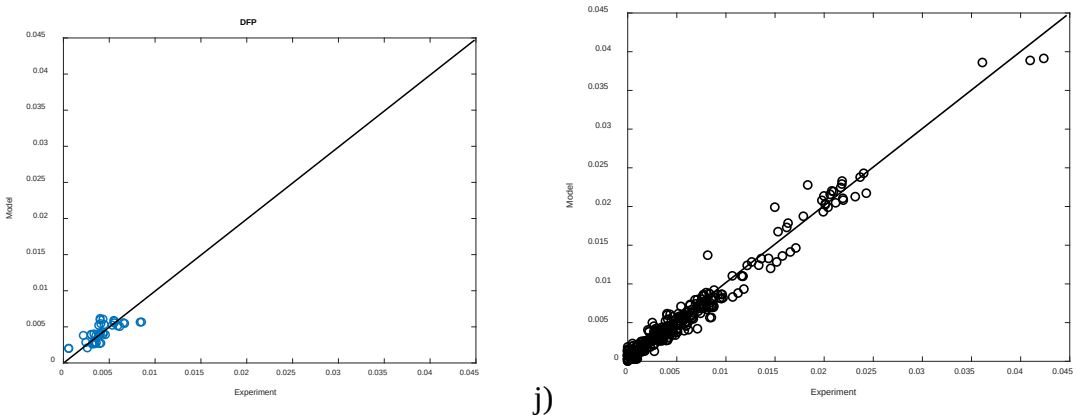

i) j)

Figure S4. Parity plots for a) citral, b) citronellal, c) isopulegol, d) neoisopulegol, e) menthol, f) neomenthol, g) neoisomenthol, h) dimethyloctanol, i) defunctionalized products, j) all products together.

4. References

[1] L. Lutterotti, Total pattern fitting for the combined size-strain-stress-texture determination in thin film diffraction, Nuclear Inst. and Methods in Physics Research, B, 268, 334-340, 2010.

[2] Wyckoff, R. W. G. Second edition. Interscience Publishers, New York, New York Crystal Structures (1963) 1, (p. 7-83).

[3] Wyckoff, R. W. G. Second edition. Interscience Publishers, New York, New York Crystal Structures (1963) 1, (p. 85-237).