Supplementary Figures

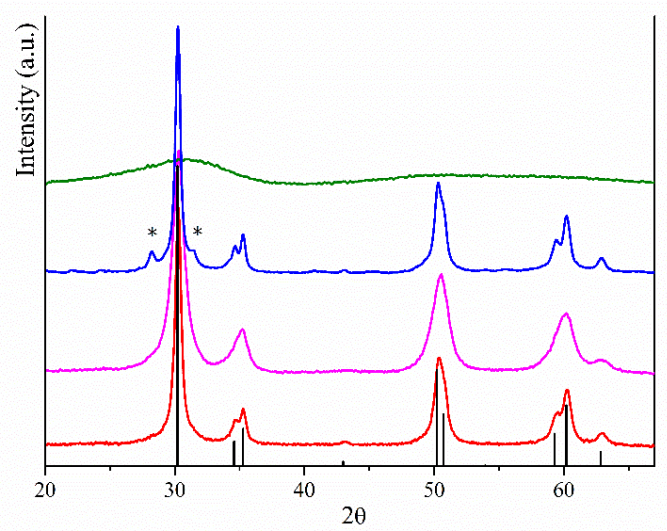


Figure S1. Experimental PXRD of Z1 in red, Z2 in blue, Z3 in pink and of the product obtained using acetic acid as hydrolysis catalyst in green compared to reference for t-ZrO_2 (ICCD card PDF n. 088-1007). Stars indicate signal of monoclinic ZrO_2 (ICCD card PDF n. 037-1484).

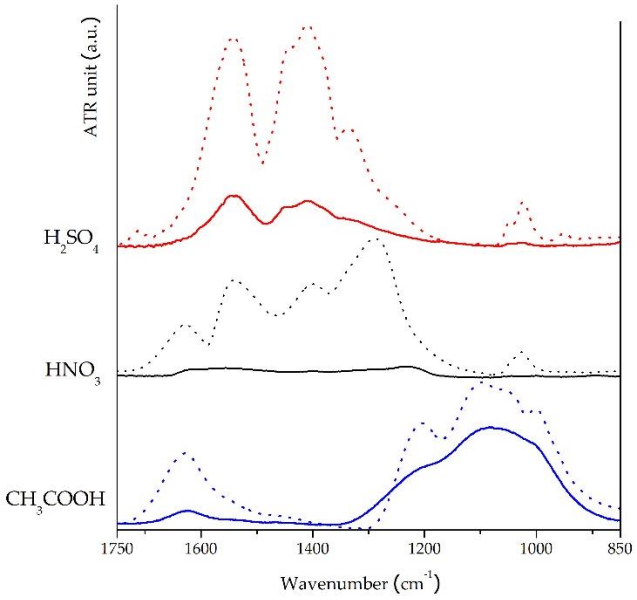
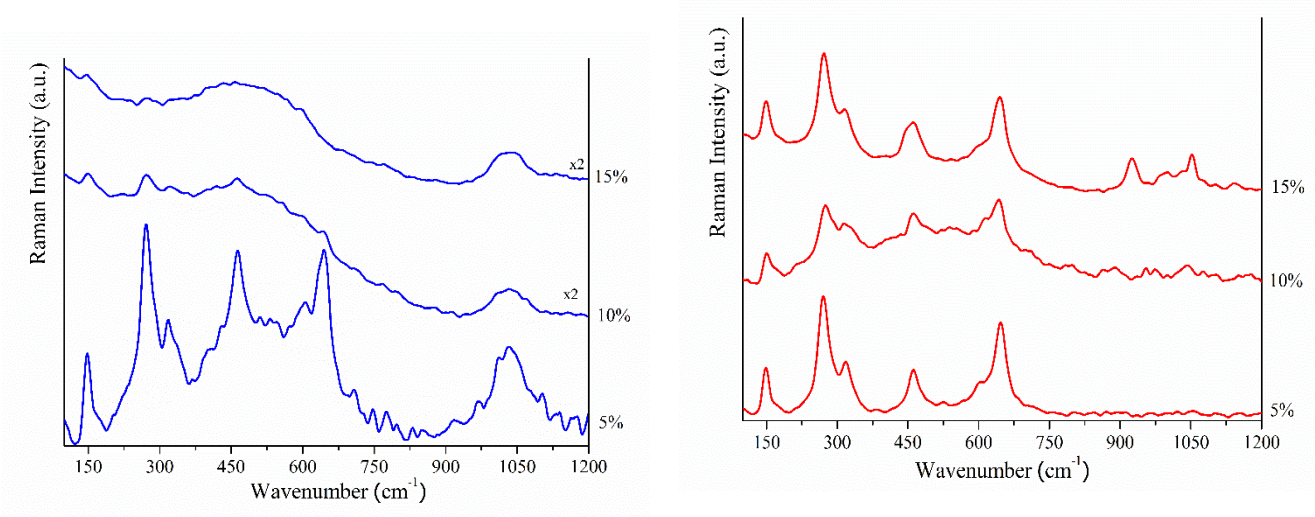
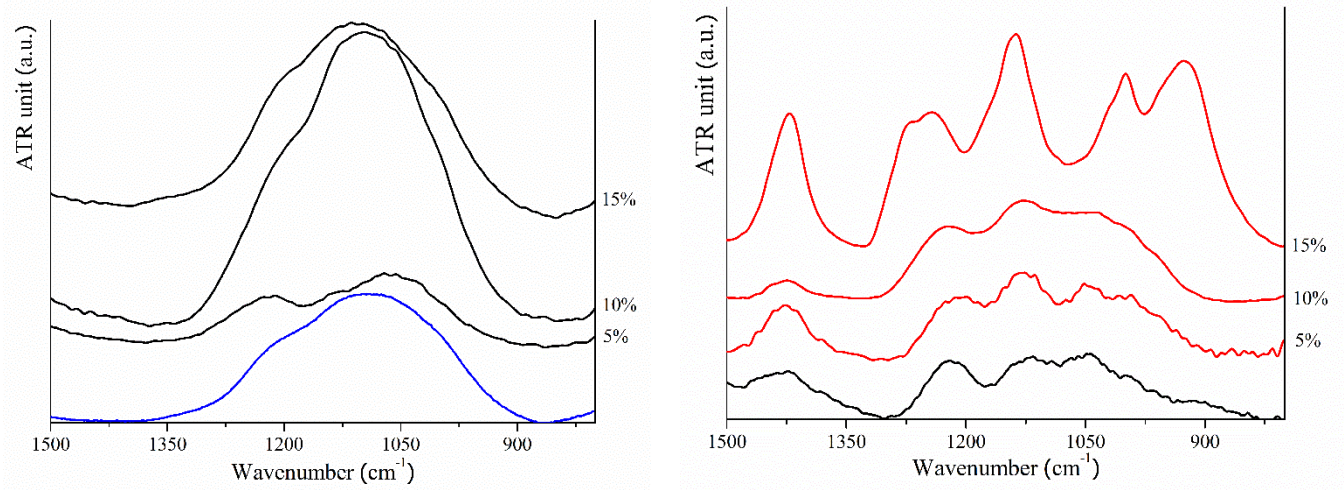


Figure S2. ATR spectra of products obtained after MW-drying (dot lines) and MW-calcination using sulfuric, nitric and acetic acid as hydrolysis catalysts (respectively in red, black and blue).



(**a**) (**b**)

Figure S3. Raman spectra of S1Z (**a**) and S2Z (**b**), impregnated by IWI with sulfates at increasing wt%.



(**a**) (**b**)

Figure S4. (**a**) ATR spectra of S1Z WI (in blue) and S1Z (in red) impregnated by IWI with sulfates at increasing wt%; (**b**) ATR spectra of S2Z_WI (in black) and S2Z (in red) impregnated by IWI with sulfates at increasing wt%.

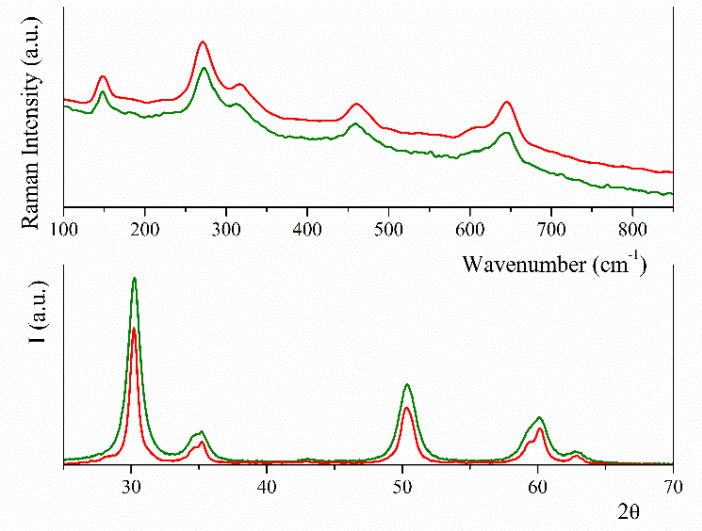
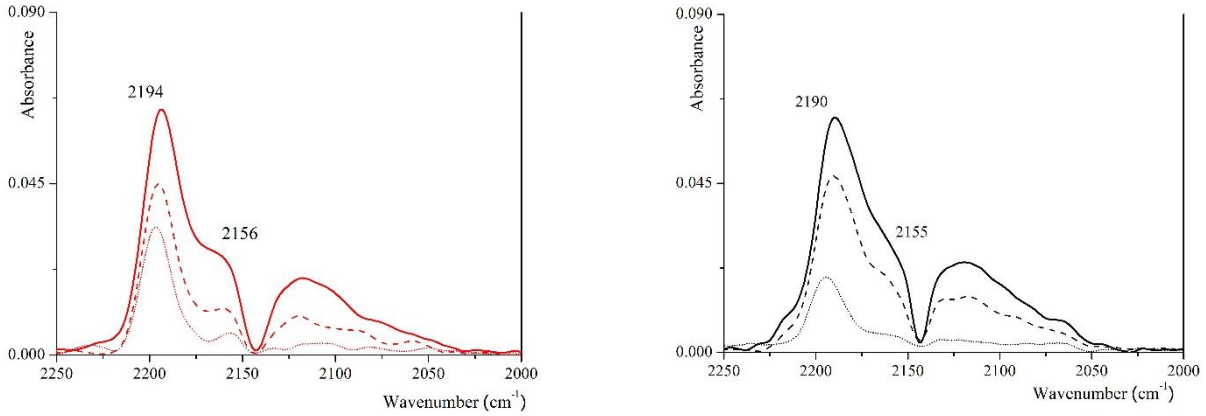


Figure S5. Raman spectrum (on the top) and powder diffractogram (on the bottom) of catalysts SZ_p (in green) and S2Z_8 (in red).



(**a**) (**b**)

Figure S6. Differential FTIR spectra of CO adsorbed on (a) S2Z_8 and (b) S1Z_5. Solid line for 100 torr, dash line for 50 torr, dot line for 5 torr.

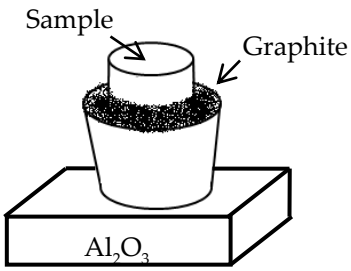


Figure S7. Schematic representation of the two crucibles set-up employed during MW-assisted calcination.

Temperature measurements during MW-assisted calcination description

In a typical MW treatment the recorded T depends on both the measurement method and the position of the measuring device within the system. Measurements can be carried out with a shielded thermocouple, a fiber optic or an infrared temperature sensor. [1] It was shown that there is often a difference in the T recognition by possible sensors. [2] In the case of the thermocouple the measure is punctual and can be done inside the reaction mixture, whereas an IR sensor usually reads temperature in the outside of the vessel, whereas a fiber optic sensor can be inserted directly in the reaction solution. Trying to define an appropriate profile temperature in our synthetic procedure, T was measured during the MW-assisted calcination using an optic pyrometer and after the treatment using a thermocouple, into both graphite and sample crucible. During the MW treatment in pulsed mode, it is possible to observe graphite powder becoming incandescent after few seconds in cycle on (red colour) and return black when irradiation stops (cycle off). Temperature variation is fast and the maximum and minimum detected T, during cycle on, were 750°C and 380°C. These values are comparable to literature data. [3] The temperature seems to be slightly higher in the center of the system, where the sample is located. It has been reported that zirconia is a MW absorber at high T [3], so we can suppose that the sample, heated by graphite, starts to absorb MW radiation. The temperature of the susceptor after MW-assisted calcination resulted to be 450°C, becoming 250°C after 5 minutes and 40°C after half an hour. Inside the sample crucible, after MW-assisted calcination, the recorded temperatures were the same. This suggests that during cycle off (of 10 s) the temperature does not get down below 450°C. We can conclude that the temperature of calcination is cyclic, reaching a maximum of 750°C at the end of cycle on and a minimum 450°C during cycle off.

1. Mirzaei, A.; Neri, G. Microwave-Assisted Synthesis of Metal Oxide Nanostructures for Gas Sensing Application: A Review. *Sens. Actuators B Chem.* **2016**, *237*, 749–775, doi:10.1016/j.snb.2016.06.114.
2. Priecel, P.; Lopez-Sanchez, J.A. Advantages and Limitations of Microwave Reactors: From Chemical Synthesis to the Catalytic Valorization of Biobased Chemicals. *ACS Sustain. Chem. Eng.* **2019**, *7*, 3–21, doi:10.1021/acssuschemeng.8b03286.
3. Bhattacharya, M.; Basak, T. A Review on the Susceptor Assisted Microwave Processing of Materials. *Energy* **2016**, *97*, 306–338, doi:10.1016/j.energy.2015.11.034.