Supplementary data

Article

Insight into active centers and anti-coke behavior of niobium-containing SBA-15 for glycerol dehydration

Katarzyna Stawicka*, Maciej Trejda and Maria Ziolek

Department of Heterogeneous Catalysis, Faculty of Chemistry, Adam Mickiewicz University, Poznan, ul. Uniwersytetu Poznanskiego 8, 61-614 Poznan; Poland; tmaciej@amu.edu.pl (M.T.); ziolek@amu.edu.pl (M.Z.);

* Correspondence: kstawick@amu.edu.pl (K.S.); Tel.:+34 618 291 794

Supplementary information

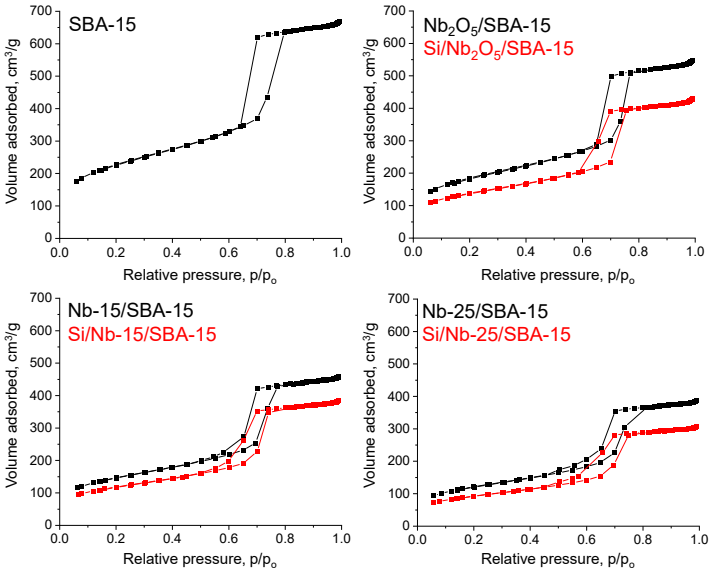

Figure S1. N_2 adsorption/desorption isotherms of SBA-15 before and after its modification with niobium species and silylation.

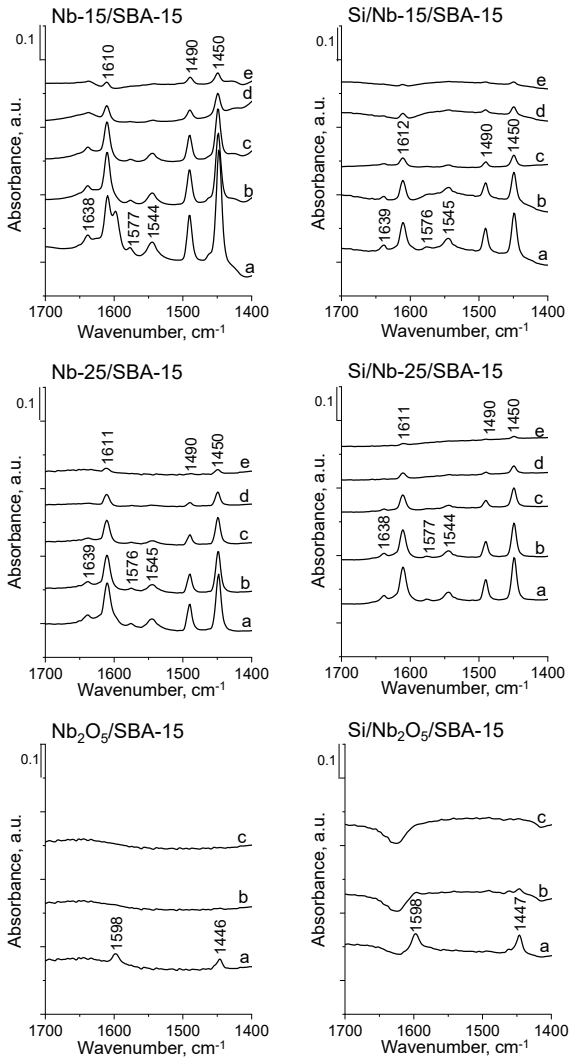

Figure S2. FTIR spectra of the samples after pyridine adsorption at 150 °C (a) and desorption at 150 – 300 °C for 30 min (150 °C (b); 200 °C (c); 250 °C (d); 300 °C (e)). The spectra of activated samples (i.e. catalysts pretreated at 400 °C in vacuum for 2 h) were subtracted from recorded spectra. Spectra were normalized to 10 mg of the catalyst.

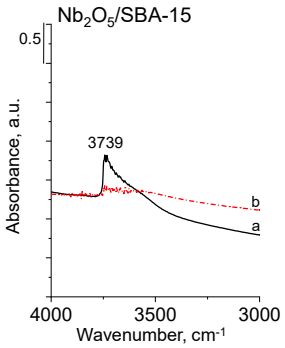

Figure S3. FTIR spectra in hydroxyl region of Nb_2O_5/SBA-15 after evacuation at 400 °C for 2h ((a) – black line) and pyridine adsorption at 150 °C ((b) – red line).

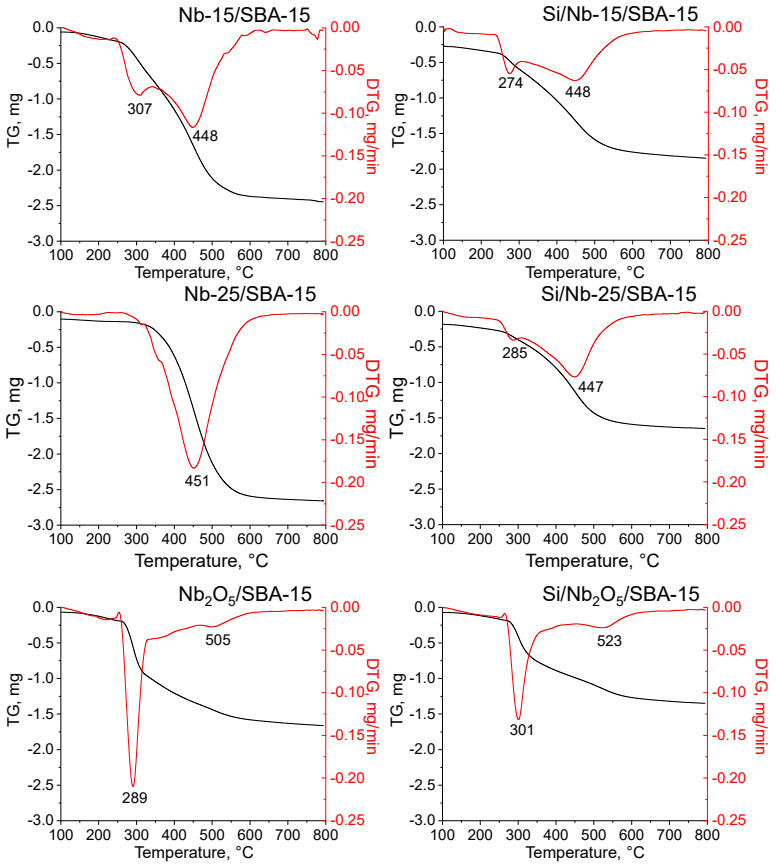

Figure S4. Thermogravimetric analyses of catalysts after 4 hours of glycerol dehydration.

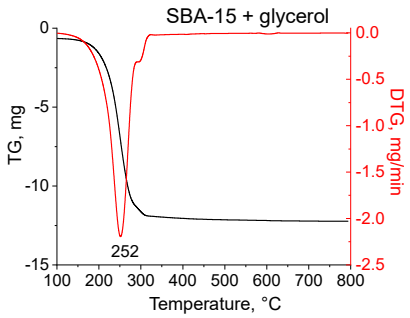

Figure S5. Thermogravimetric analysis of SBA-15 material mixed with glycerol.

Table S1. Results of glycerol dehydration. Conditions: mass of catalyst: 100 mg; catalyst pretreating: 2 h 400 °C in argon flow; reaction temperature: 350 °C; speed of glycerol dosing: 1 mL/h; Ar flow: 50 mL/min. Conversion of glycerol calculated from the total yield of products.

Catalyst	time, h	Conversion, %	Yield (Selectivity), %					
			acetaldehyde	acrolein	acetone	acetic acid	hydroxyacetone	coke deposit/h*
SBA-15	1	4.5	0.2 (3.4)	0	0	0	0	4.3 (96.6)
	2	4.5	0.2 (3.4)	0	0	0	0	
	3	4.5	0.2 (3.4)	0	0	0	0	
	4	4.5	0.2 (3.4)	0	0	0	0	
Nb-15/SBA-15	1	41.7	0.9 (2.2)	9.1 (21.8)	0.2	0.3	0.2 (0.5)	31.0 (74.2)
	2	42.3	0.7 (1.7)	9.7 (22.9)	0.2	0.2	0.5 (1.2)	
	3	42.0	0.6 (1.4)	9.5 (22.6)	0.2	0.1	0.6 (1.4)	
	4	41.3	0.6 (1.5)	8.8 (21.3)	0.2	0.1	0.6 (1.5)	
Nb-25/SBA-15	1	95.5	4.1 (4.3)	43.0 (45.0)	1.3	1.6	1.6 (1.7)	43.9 (47.4)
	2	100	3.8 (3.8)	43.5 (44.5)	1.4	1.0	2.9 (2.9)	
	3	89.4	3.6 (4.0)	38.5 (43.1)	1.3	0.7	1.4 (1.6)	
	4	85.9	3.2 (3.7)	34.2 (39.8)	1.1	0.7	2.8 (3.3)	
Nb_2O_5/SBA-15	1	24.5	1.3 (5.3)	12.8 (52.2)	0.1	0	0.8 (3.3)	9.5 (41.1)
	2	23.4	1.3 (5.5)	11.7 (50.0)	0.1	0	0.8 (3.4)	
	3	22.9	1.4 (6.1)	11.2 (48.9)	0.1	0	0.7 (3.1)	
	4	21.9	1.2 (5.5)	10.6 (48.4)	0.1	0	0.5 (2.3)	
Si/Nb-15/SBA-15	1	30.3	0.7 (2.3)	9.8 (32.3)	0.1	0.1	0.4 (1.3)	19.2 (67.6)
	2	28.0	0.4 (1.4)	7.7 (27.5)	0.1	0.1	0.5 (1.8)	
	3	27.9	0.4 (1.4)	7.7 (27.6)	0.1	0	0.5 (1.8)	
	4	27.2	0.3 (1.1)	7.2 (26.5)	0.1	0	0.4 (1.5)	
Si/Nb-25/SBA-15	1	78.3	4.5 (5.7)	50.4 (64.4)	0.5	0.9	2.7 (3.4)	19.3 (27.5)
	2	68.8	3.4 (4.9)	41.1 (61.3)	0.6	0.5	3.9 (5.7)	
	3	69.7	3.2 (4.6)	42.3 (60.7)	0.6	0.3	4.0 (5.7)	
	4	63.9	2.4 (3.8)	38.1 (59.6)	0.3	0.4	3.4 (5.3)	
Si/Nb_2O_5/SBA-15	1	14.5	0.9 (6.2)	5.8 (40.0)	0.1	0	0.3 (2.1)	7.4 (60.7)
	2	13.0	0.8 (6.2)	4.4 (33.8)	0.1	0	0.3 (2.3)	
	3	10.8	0.6 (5.6)	2.5 (23.1)	0.1	0	0.2 (1.8)	
	4	10.3	0.9 (8.7)	1.7 (16.5)	0.1	0	0.2 (1.9)	

*coke deposit was analyzed after 4 h of the reaction and was evenly distributed for each hour