

Synthesis and physico-chemical characterization of Beta-Bentonite composite materials for shaped catalysts

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Supporting Information

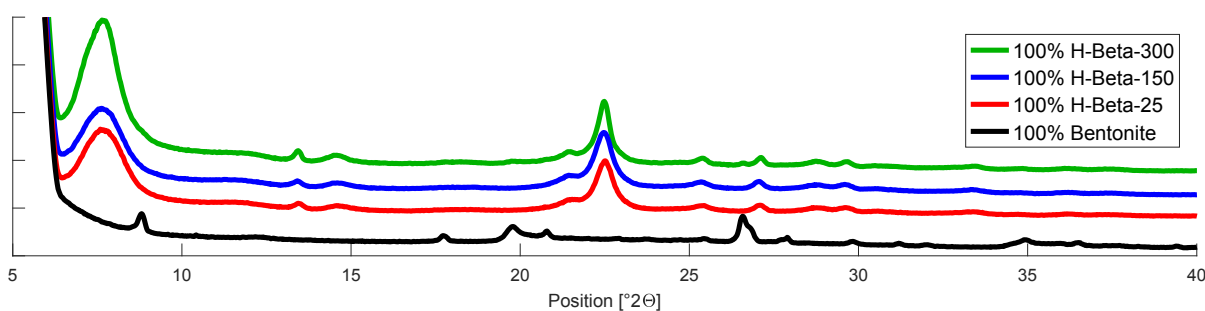


Figure S1. XRD patterns of pristine H-Beta-25, H-Beta-150, H-Beta-300 zeolites and bentonite.

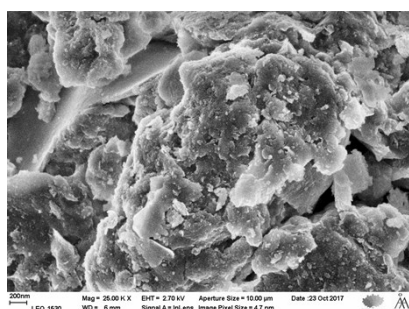


Figure S2. SEM image: Morphology of pristine bentonite.

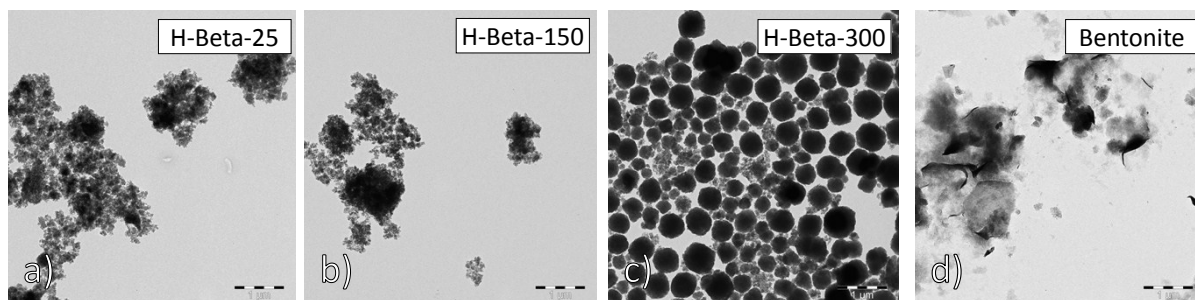


Figure S3. TEM images of pristine materials. Scale bar 1 μm .

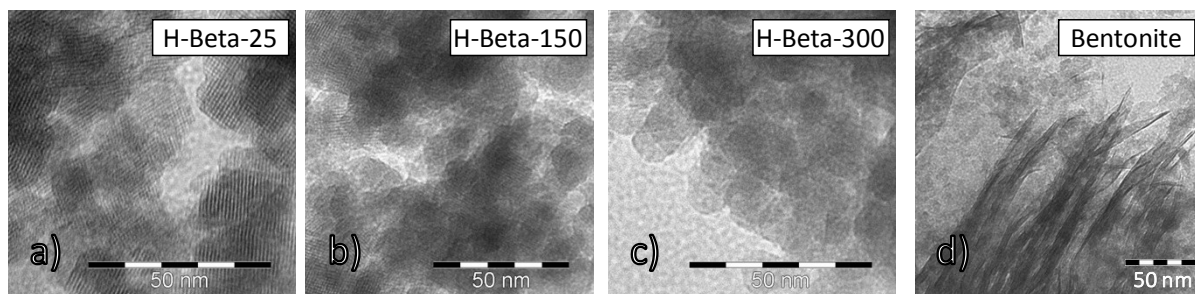


Figure S4. TEM images of pristine materials. Scale bar 50 nm.

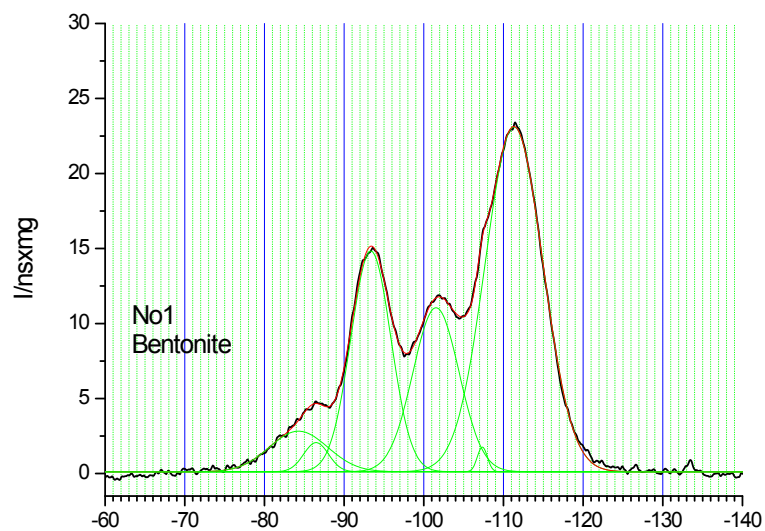


Figure S5. ^{29}Si MAS NMR spectrum for Bentonite sample. The spectrum (black line) is fitted with six Gaussian lines (green lines, the sum is a red line). The parameters of the component lines are given in Table S2.

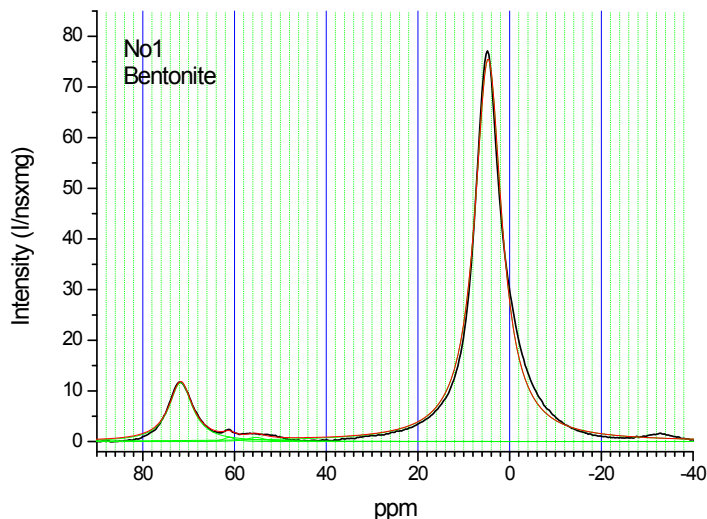


Figure S6. ^{27}Al MAS NMR spectrum of the Bentonite sample. The spectrum has been fitted with 4 Lorentzian lines, with parameters given in Table S3.

Table S1 Elemental analysis of pristine H-Beta-25, H-Beta-150, H-Beta-300 zeolites and bentonite.

Zeolite	Binder	SiO ₂	Al ₂ O ₃	MgO	K ₂ O	Fe ₂ O ₃	SiO ₂ /Al ₂ O ₃	
-	wt. %	wt. %	wt. %	wt. %	wt. %	wt. %	wt/wt	mol/mol
H-Beta-25	0	93.6	6.4	0.0	0.0	0.0	15	25
H-Beta-150	0	93.3	6.7	0.0	0.0	0.0	14	23
H-Beta-300	0	98.4	1.6	0.0	0.0	0.0	60	102
Bentonite	100	78.1	15.4	1.9	1.5	3.1	5	9

Table S2. Frequency shift, Gaussian width, relative area and total area of the components in ^{29}Si MAS NMR spectra of studied samples*.

Sample	No1 Bentonite	No2 B25B10	No3 B25B20	No4 B25B30	No5 B25B65	Assignment
Peak1 (ppm)	-111.3	-115.3	-115.2	-115.3	-115.3	Beta sites
width (ppm)	7.2	2	2.1	2.1	2	
Area (%)	50.2	7.5	7.2	6.8	3.6	
Peak2 (ppm)	-107.4	-111.2	-111.2	-111.3	-111.3	Beta sites
width (ppm)	1.3	2.9	2.9	2.9	2.8	
Area (%)	0.6	37.8	34.6	33.2	19.1	
Peak3 (ppm)	-101.5	-102.7	-102.7	-102.7	-103	Q ³
width (ppm)	5.8	2.9	2.8	3	3.1	
Area (%)	19.1	5	4.4	4.4	2.9	
Peak4 (ppm)	-93.4	-98.4	-99.2	-98.3	-99.6	Q ³ (1Al)
width (ppm)	5	7	6.7	6.7	7	
Area (%)	22.3	6.1	6.9	7.6	11.3	
Peak5 (ppm)	-86.5		-90.8	-89.9	-93	unresolved
width (ppm)	3.1		7	7	12.6	
Area (%)	1.8		1.8	2.3	11.9	
Peak6 (ppm)	-84.3	-110	-110	-109.8	-109.9	unresolved

width (ppm)	7.3	9	9.2	9.5	9
Area (%)	6	43.6	45.1	45.8	51.1
Total Area	415	828	782	759	603

B25B10: 90% H-Beta 25 +10% Bentonite; B25B20: 80% H-Beta-25 +20% Bentonite; B25B30: 70% H-Beta-25 +30% Bentonite; B25B65: 35% H-Beta-25 + 65% Bentonite.

Table S3. Frequency shift, Lorentzian width, relative area and total area of the components in ^{27}Al MAS NMR spectra of studied samples*.

Sample	No1 Bentonite	No2 B25B10	No3 B25B20	No4 B25B30	No5 B25B65	Assignment
Peak1 (ppm)	61.1	58.3	58.2	58.3	58.9	Al IV of Beta
width (ppm)	3	3.6	4.9	5.3	7.7	
Area (%)	0.4	7.2	11.5	9.1	7.3	
Peak2 (ppm)	55.3	54.2	54.2	54.3	54.4	Al IV of Beta
width (ppm)	4.7	4.5	4.4	4.5	5.3	
Area (%)	0.6	40.9	34.5	26.6	13.5	
Peak3 (ppm)	4.7	2.3	3.3	3.6	3.9	Al VI of clay
width (ppm)	7.1	9.8	9.5	8.8	8.9	
Area (%)	87.3	21.0	22.3	25.2	31.1	
Peak4 (ppm)		-0.1	-0.1	-0.1	-0.3	Al VI of beta
width (ppm)		1.7	1.6	1.6	1.6	
Area (%)		2.6	2.1	1.8	0.7	
Peak5 (ppm)		-4.4	-4.4	-4.2	-3.7	Al VI of beta
width (ppm)		5.4	5.6	6.2	5.2	
Area (%)		6.0	6.5	5.2	1.9	
Peak6 (ppm)		-9.1	-9.0	-8.9	-7.9	Al VI
width (ppm)		4.7	4.9	5.6	4.6	
Area (%)		2.7	2.5	2.1	0.8	
Peak7 (ppm)		47.6	48.0	48.7	49.1	Al IV
width (ppm)		9.4	7.9	9.1	7.1	
Area (%)		5.3	3.5	3.4	2.0	
Peak8 (ppm)		25.3	23.7	24.5	23.7	Al V
width (ppm)		24.1	26.9	29	29.2	
Area (%)		14.2	16.9	22.7	35	
Peak9 (ppm)		-	69.2	68	65.9	
width (ppm)		-	6.9	9.6	7	
Area (%)		-	1.1	3.9	6	
Peak10 (ppm)	71.7				71.4	
width (ppm)	6.2				4.1	
Area (%)	11.7				1.7	
Total Area	968	848	922	965	1015	

B25B10: 90% H-Beta 25 +10% Bentonite; B25B20: 80% H-Beta-25 +20% Bentonite; B25B30: 70% H-Beta-25 +30% Bentonite; B25B65: 35% H-Beta-25 + 65% Bentonite.

Table S4. Frequency shifts, Gaussian width integral intensities and assignment of the resonances in the ^{29}Si MAS NMR spectrum of bentonite.

	Frequency shift (ppm)	Gaussian width (ppm)	Area	%	Assignment
Peak1(N)	-111.33	7.24	208.45	50.2	Q ⁴ amorphous

o1)					%	
Peak2(N					0.6	Q ⁴ , quartz
o1)	-107.35	1.25	2.57		%	
Peak3(N					19.1	Q ³ amorphous
o1)	-101.54	5.80	79.43		%	
Peak4(N					22.3	Q ³ _{clay}
o1)	-93.36	5.03	92.71		%	
Peak5(N					1.8	Q ³ _{clay} (1Al)
o1)	-86.48	3.10	7.55		%	
Peak6(N					6.0	Q ² , Q ³ _{clay} (1Al)
o1)	-84.28	7.31	24.77		%	
			Sum:		100.	
			415.48		0%	

Table S5. Parameters of the Lorentzian lines in ²⁷Al spectrum of Bentonite.

Bentonite	Frequency shift, ppm	width ppm	Area abs	Area %	Assignment
Peak1	4.7	7.1	845.3	87.3 %	Al VI
Peak2	71.7	6.2	112.9	11.7 %	Al IV
Peak3	55.3	4.7	6.1	0.6%	Al IV
Peak4	61.1	3.0	4.0	0.4%	Al IV
		Sum:	968.2	100.0 %	