Preparation of ordered large pore SBA-15 silica functionalized with aminopropyl-group through one-pot synthesis

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[†]Electronic Supplementary Information including Pore size distributions, TEM, ²⁹Si {¹H} CPMAS NMR spectra, TGA and the results of Knoevenagel reaction of carbonyl compounds with ethyl cyanoacetate



Fig. 1 Nitrogen adsorption-desorption isotherms and BJH pore size distribution plot (inset) of the extracted materials with different amounts of aminopropyl groups.

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100 nm (c) (d)

Fig. 2 Transmission electron micrographs of the extracted SBA-15 mesoporous silica functionalized with various amounts of aminopropyl groups: (a) 0 %, (b) 10 %, (c) 15 % and (d) 20 %.



δ (ppm)

Fig. 3 ²⁹Si $\{^{1}H\}$ CPMAS NMR spectra of extracted functionalized with different amounts of aminopropyl groups in the initial mixture: (a) 5 %, (b) 10 %, (c) 15 %, and (d) 20 %.



Fig. 4. TGA and DTG profiles of the as-synthesized (dash line) and the extracted amino-functionalized SBA-15 with 10% aminopropyl groups in the synthesis mixture.

Table 3. Knoevenagel reaction of carbonyl compounds (10 mmol) with ethyl cyanoacetate (10 mmol) in cyclohexane (10 mL) over extracted amino-functionalized SBA-15 (0.15 g) with 10% aminopropyl groups in the initial mixture.

	$R \rightarrow 0 + CO_2 $	\xrightarrow{Et} \xrightarrow{R} $\xrightarrow{R'}$	CO ₂ Et + H ₂ O	
Reaction	R	R'	Time (h)	Yield [*] (%)
1	C_6H_5	Н	1	>99
2	C_4H_9	Me	8	89.3
3	Et	Et	8	69.4
4	C_3H_7	Et	8	69.6
5	cC_5H_{10}	-	3	>99
6	C_6H_5	Me	24	50.3

^{*} The product was identified by GC-MS and analyzed by GC, using n-dodecane as internal standard. The removal of water was not carried out in the reaction procedure.