

*Supporting information for*  
**Optimization of ammonium fluoride route to hierarchical zeolites**

T. Onfroy<sup>1,2\*</sup>, Z. Qin<sup>2</sup>, S. Casale<sup>1</sup>, V. Valtchev<sup>2\*</sup>

<sup>1</sup> Sorbonne Université, CNRS, Laboratoire de Réactivité de Surface, F-75005 Paris, France

<sup>2</sup> Normandie Université, ENSICAEN, UNICAEN, CNRS, LCS, 14050 Caen, France

**\*Corresponding authors**

Dr. Thomas Onfroy, E-mail: [thomas.onfroy@sorbonne-universite.fr](mailto:thomas.onfroy@sorbonne-universite.fr)

Dr. Valentin Valtchev, E-mail: [valentin.valtchev@ensicaen.fr](mailto:valentin.valtchev@ensicaen.fr)

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Figure S2: <sup>27</sup>Al MAS NMR spectra of the parent NH<sub>4</sub>-ZSM-5 and of samples etched in 20 g of a 40 wt% NH<sub>4</sub>F solution for 0.5; 1.0; 1.5 and 2.0 h. Amount of zeolite used: 1 g (a), 2 g (b) and 4 g (c).

Figure S3: N<sub>2</sub> adsorption isotherms (a, c, e) and BJH pore size distribution (b, d, f) of the parent NH<sub>4</sub>-ZSM-5 and the samples etched in 20 g of a 40 wt% NH<sub>4</sub>F solution for 0.5; 1.0 and 2.0 h. Amount of zeolite used: 1 g (a, b), 2 g (c, d) and 4 g (e, f).

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Figure S6: TEM monitoring of the dissolution of MFI as a function of the concentration of NH<sub>4</sub>F (wt%) etching solution for 2h treatment: (a) NH<sub>4</sub>F(6)-ZSM-5(1)-2; (b) NH<sub>4</sub>F(4)-ZSM-5(1)-2; (c) NH<sub>4</sub>F(2)-ZSM-5(1)-2; (d) NH<sub>4</sub>F(1)-ZSM-5(1)-2.

Table S1: Initial F/(Al+Si) atomic ratio of the NH<sub>4</sub>F/ZSM-5/water suspension, maximum yield of the etching and Si/Al atomic ratio and porosity of parent and NH<sub>4</sub>F treated ZSM-5.

**Sample codes:** In the following, the samples will be denote: NH<sub>4</sub>F(X)-ZSM-5(Y)-Z where X and Y respectively stand for the weight of NH<sub>4</sub>F and NH<sub>4</sub>-ZSM-5 in gram per 12 g of water and Z correspond to the reaction duration in hour.

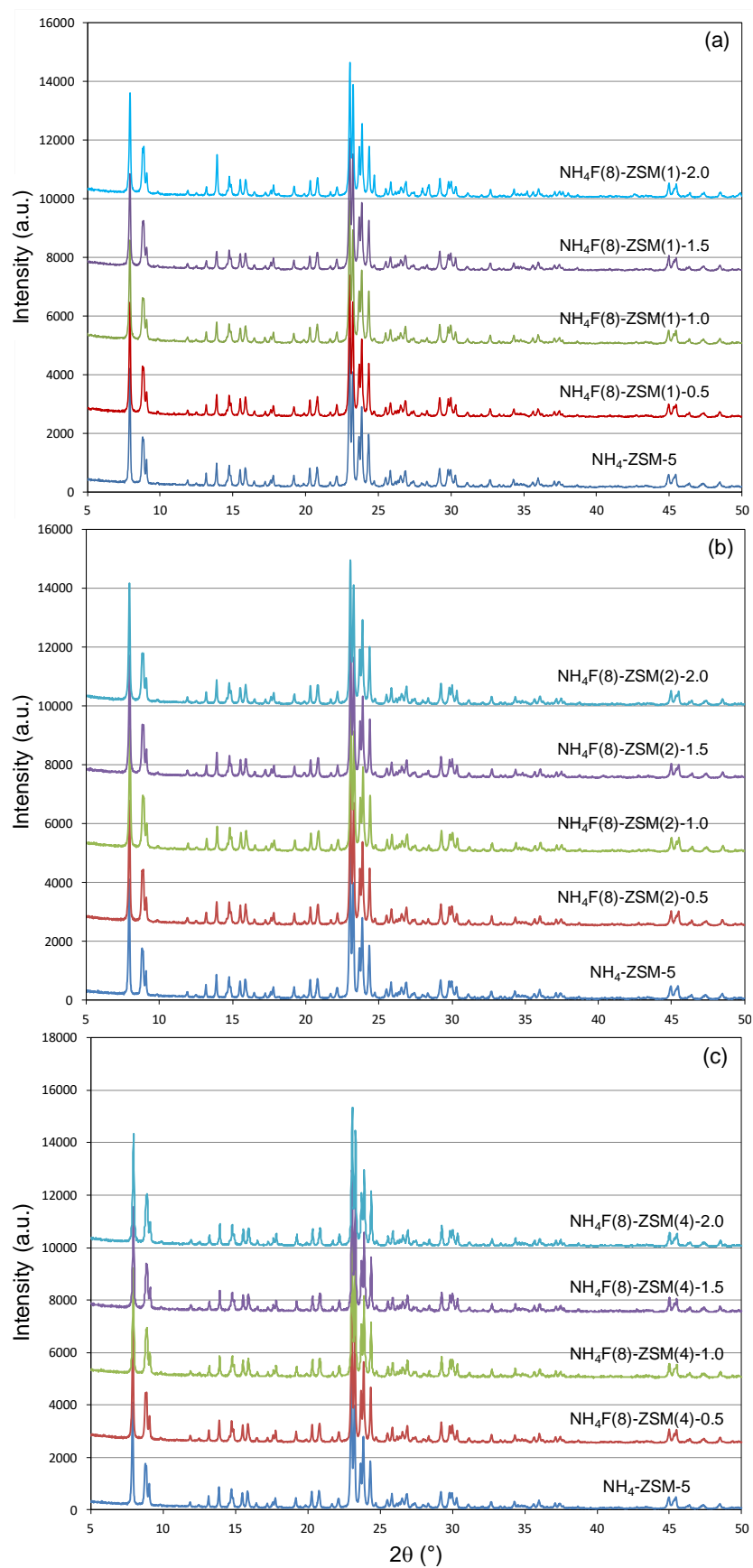


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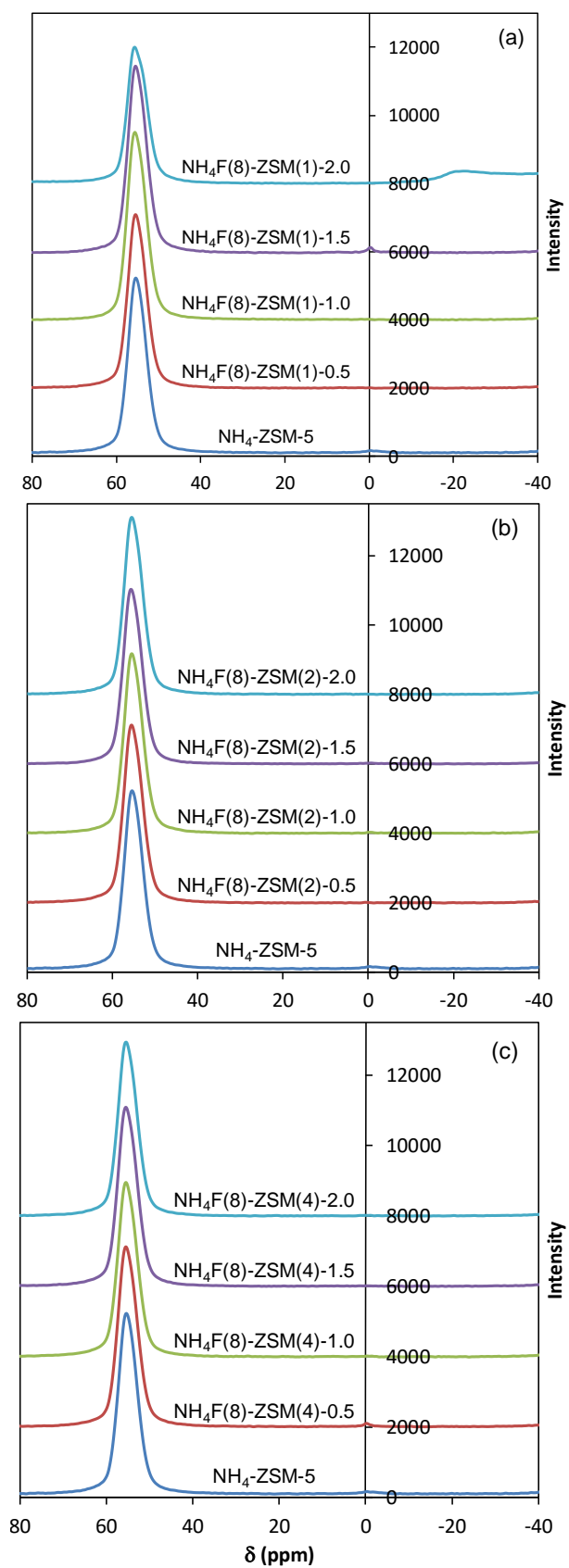


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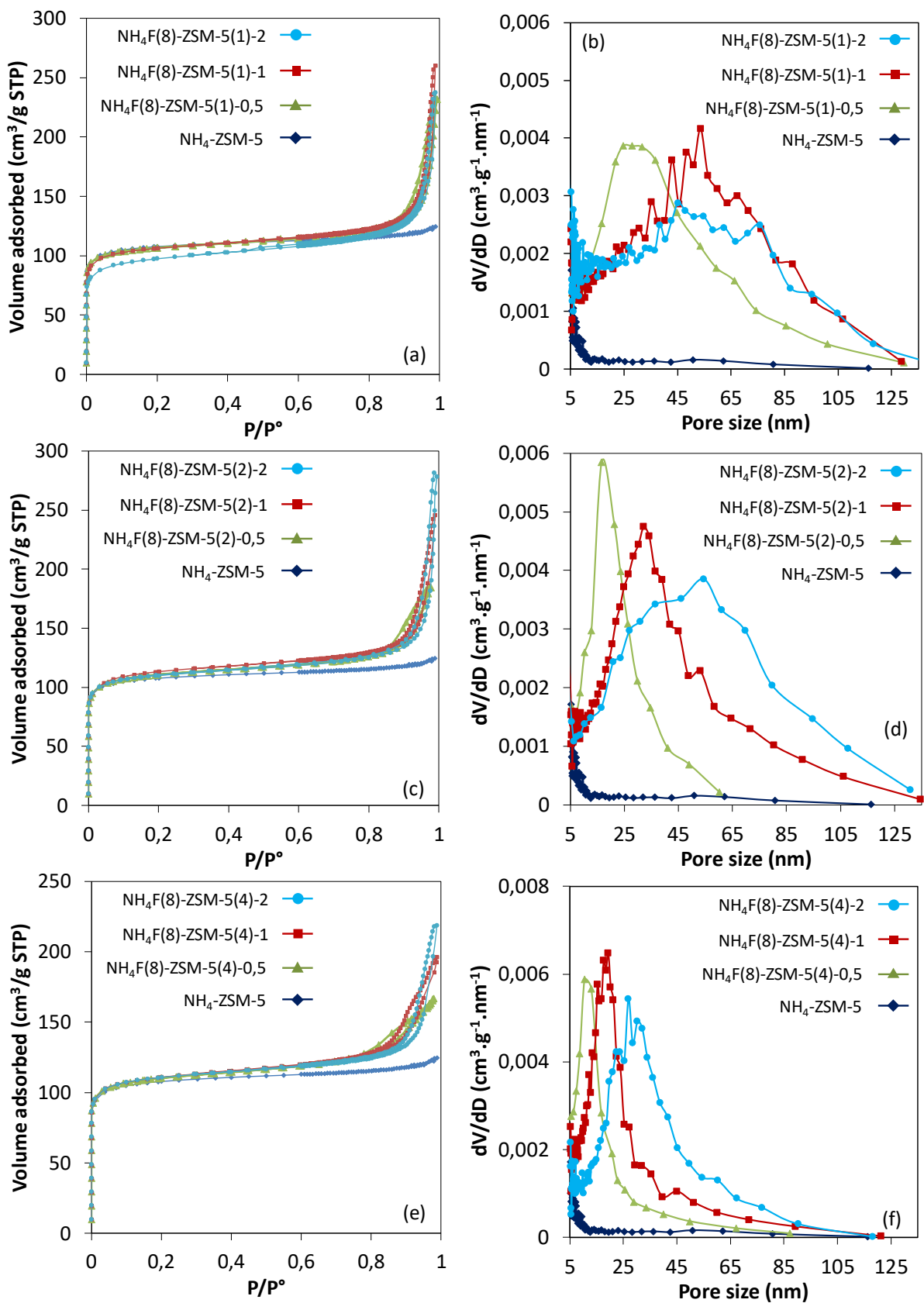


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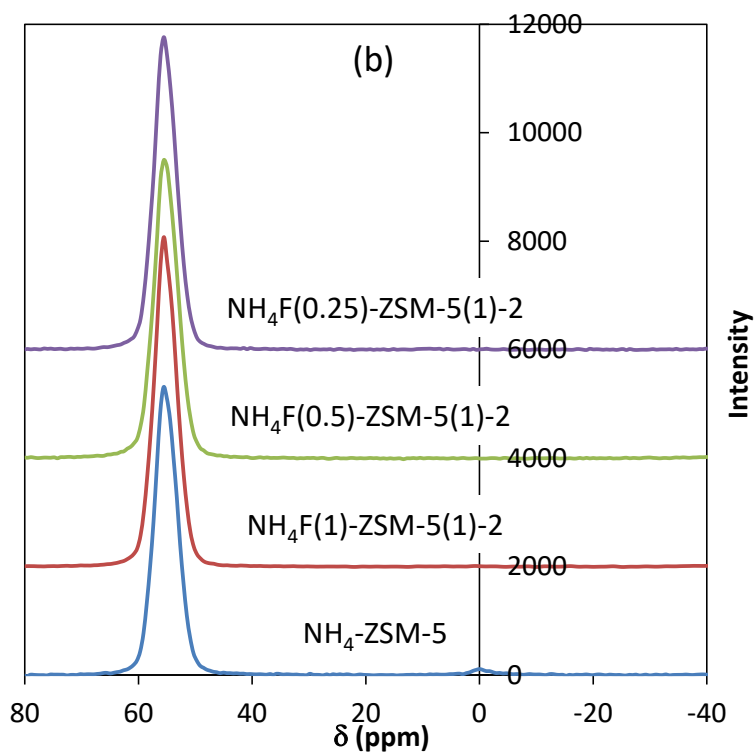
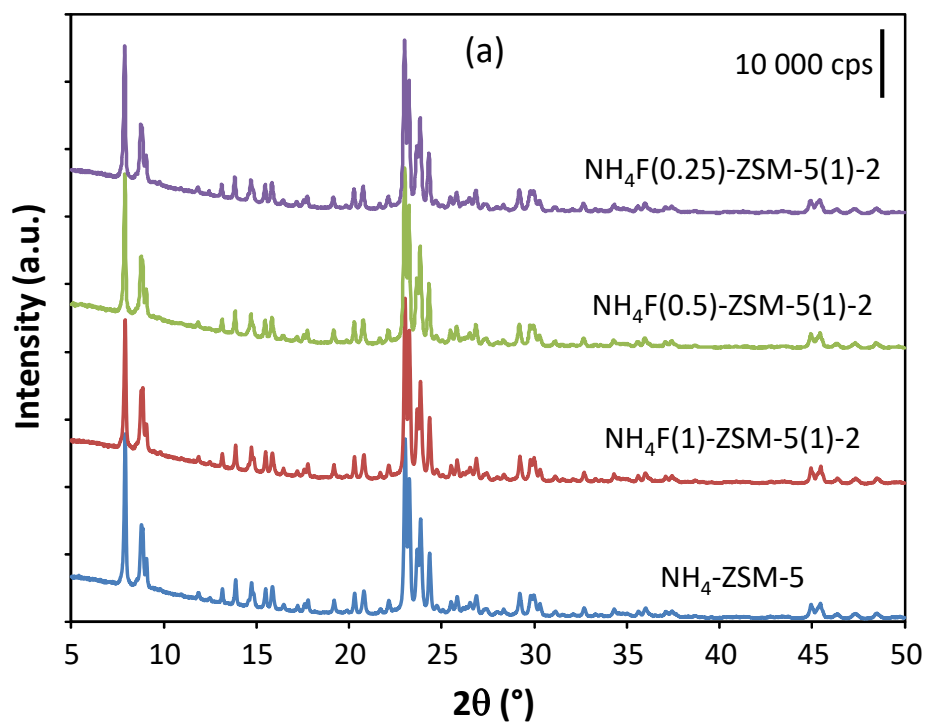


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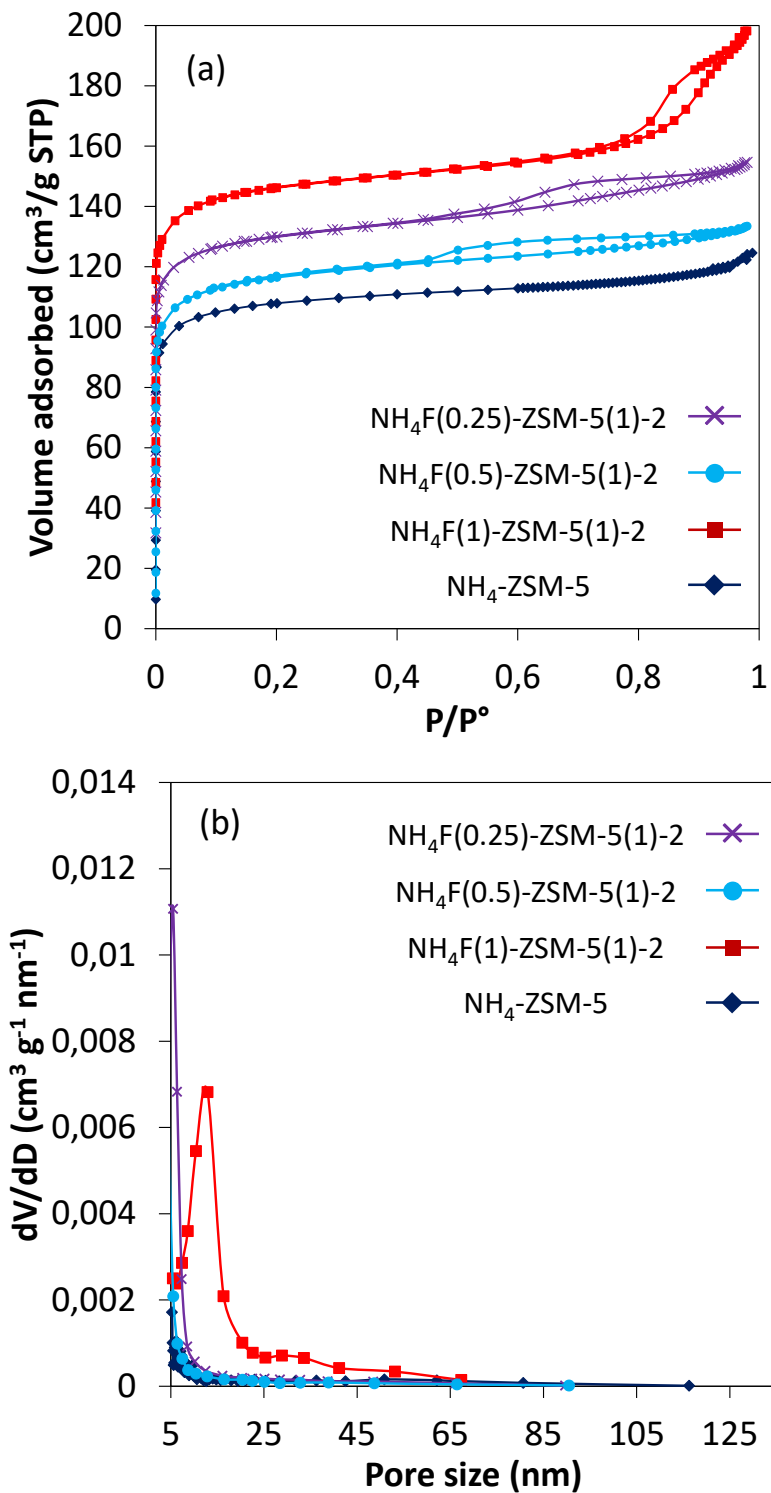


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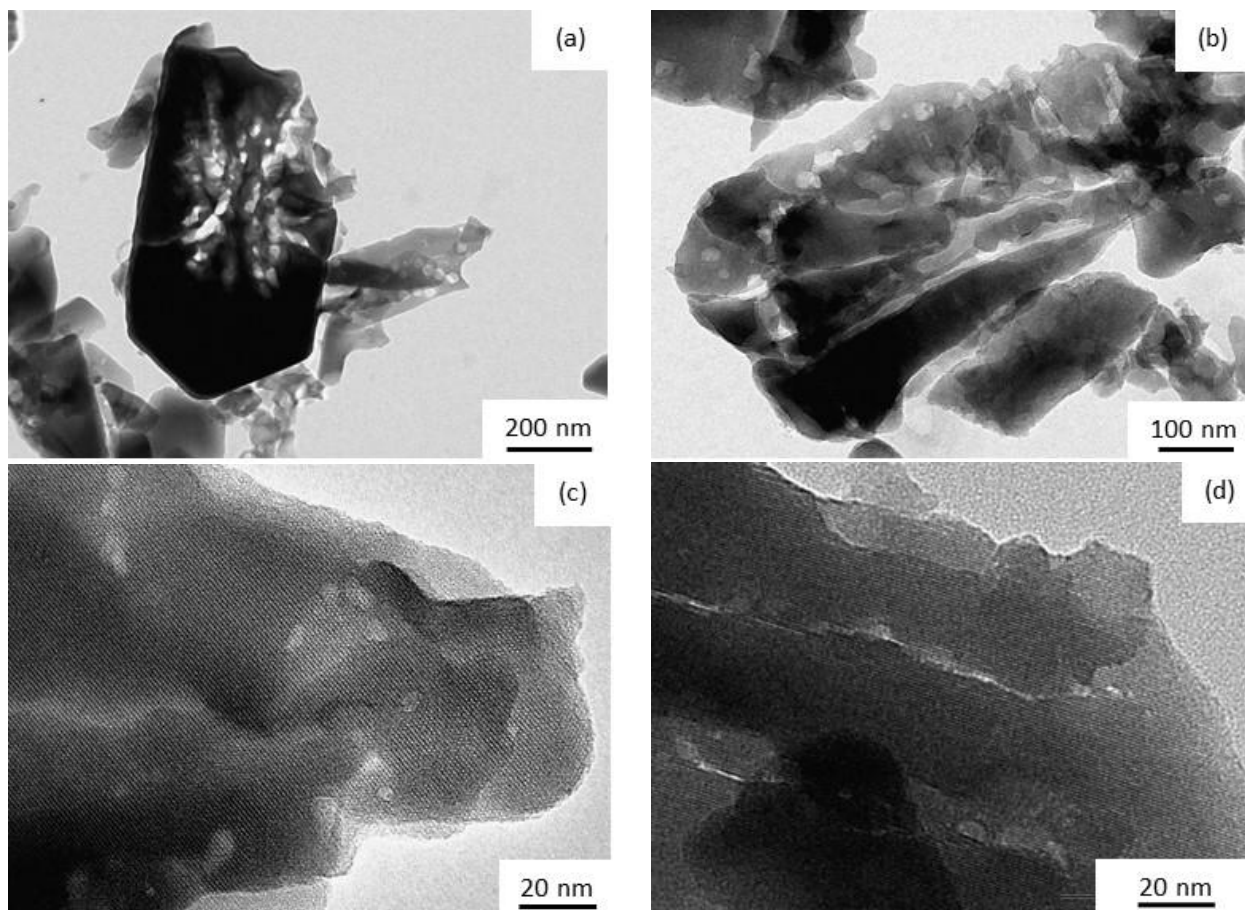


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Table S1: Initial F/(Al+Si) atomic ratio of the NH<sub>4</sub>F/ZSM-5/water suspension, maximum yield of the etching and Si/Al atomic ratio and porosity of parent and NH<sub>4</sub>F treated ZSM-5.

Samples	F/(Al+Si) <sub>ini</sub> <sup>a</sup>	% Y <sub>max</sub>	Si/Al <sup>b</sup>	S <sub>BET</sub> m <sup>2</sup> g <sup>-1</sup>	S <sub>Ext</sub> <sup>c</sup> m <sup>2</sup> g <sup>-1</sup>	V <sub>micro</sub> <sup>c</sup> cm <sup>3</sup> g <sup>-1</sup>	V <sub>meso</sub> <sup>d</sup> cm <sup>3</sup> g <sup>-1</sup>
NH <sub>4</sub> -ZSM-5	-	-	17	325	14	0.16	0.03
NH <sub>4</sub> F(8)-ZSM-5(1)-0.5	12.4	-	16	345	35	0.15	0.19
NH <sub>4</sub> F(8)-ZSM-5(1)-1		-	18	332	37	0.15	0.25
NH <sub>4</sub> F(8)-ZSM-5(1)-2		10	13	299	40	0.14	0.23
NH <sub>4</sub> F(8)-ZSM-5(2)-0.5	6.2	-	18	342	37	0.16	0.13
NH <sub>4</sub> F(8)-ZSM-5(2)-1		-	18	345	35	0.16	0.22
NH <sub>4</sub> F(8)-ZSM-5(2)-2		25	15	355	37	0.15	0.28
NH <sub>4</sub> F(8)-ZSM-5(4)-0.5	3.1	-	14	340	40	0.15	0.10
NH <sub>4</sub> F(8)-ZSM-5(4)-1		-	14	336	37	0.16	0.15
NH <sub>4</sub> F(8)-ZSM-5(4)-2		60	14	360	31	0.16	0.18