Supplementary material

Acylation of Anisole With Benzoyl Chloride Over Rapidly Synthesized Fly Ash–Based HBEA Zeolite

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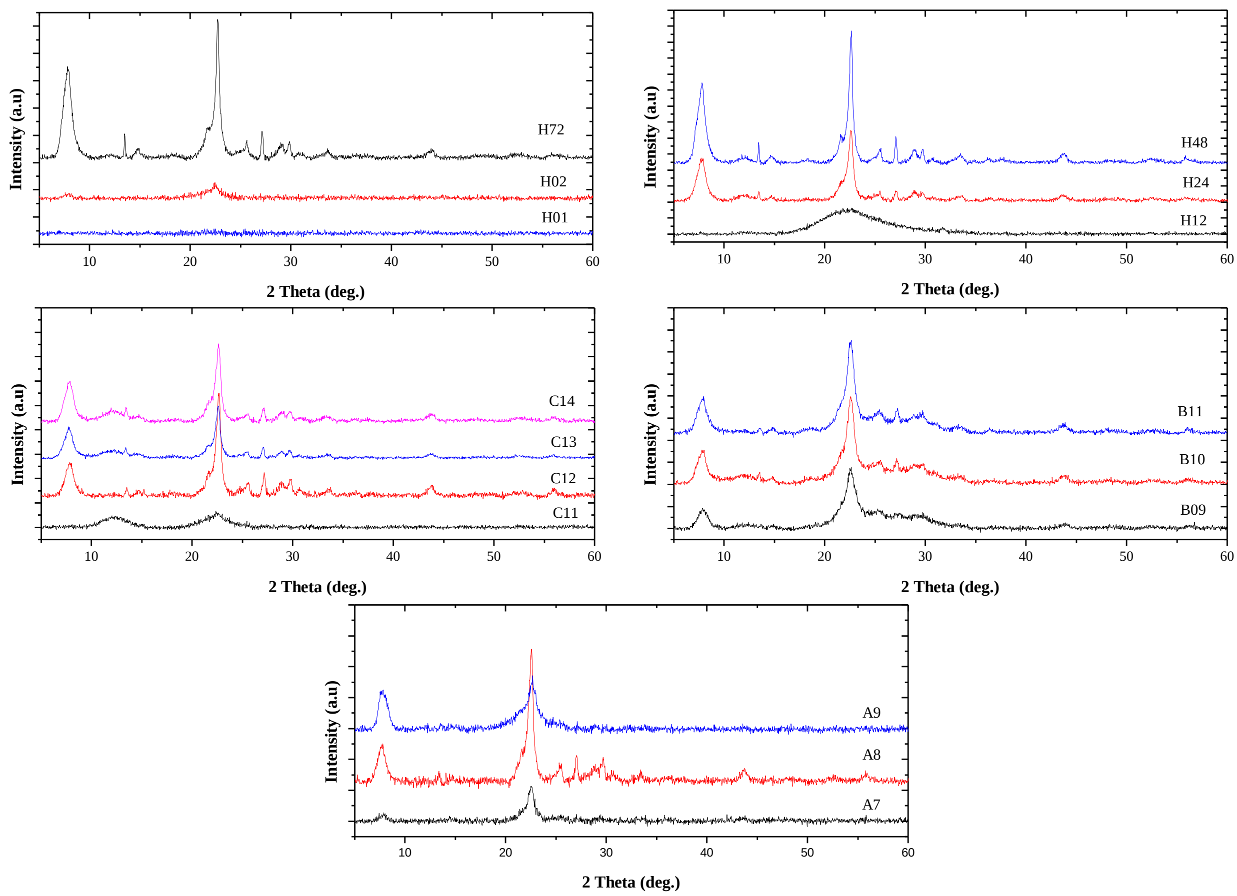
Supplementary Table S1. Molar composition for the synthesis mixture and phase formation at applied conditions

Code name	[a]Molar ratio of synthesis mixture					Synthesis time, h	[b]Phase formation
	Si	Al	Na	TEAOH	H_2O		
H01	1	0.017	0.241	0.399	17.958	72	AMR
H02	1	0.017	0.241	0.399	12.828	72	AMR+BEA
H72	1	0.017	0.241	0.399	8.980	72	BEA
H48	1	0.017	0.241	0.399	8.980	48	BEA
H24	1	0.017	0.241	0.399	8.980	24	BEA
H12	1	0.017	0.241	0.399	8.980	12	AMR
C11	1	0.017	0.241	0.399	5.986	12	AMR
C12	1	0.017	0.241	0.399	3.991	12	BEA
C13	1	0.017	0.241	0.399	2.661	12	BEA
C14	1	0.017	0.241	0.399	1.776	12	BEA
B09	1	0.017	0.241	0.399	3.991	10	BEA
B10	1	0.017	0.241	0.399	2.661	10	BEA
B11	1	0.017	0.241	0.399	1.776	10	BEA
A7	1	0.017	0.241	0.399	3.991	8	BEA
A8	1	0.017	0.241	0.399	2.661	8	BEA
A9	1	0.017	0.241	0.399	1.776	8	BEA

[a]Calculated from the metal oxide as determined by XRF of the nanosilica and the amount of NaOH or Al added.
[b]AMR means an amorphous phase in the as-synthesised product as determined by XRD.



Supplementary Figure S1. XRD patterns showing the effect of reduced molar quantities of water in the synthesis mixture on the hydrothermal time and phase purity of BEA zeolites (see Table 1).

Supplementary Table S2. Detailed ^{27}Al MAS NMR spectroscopic data after FAl and EFAl deconvolution in HBEA samples.

Sample	**Framework Al**					**Extra-framework Al**		
	$^1T_{Al}$	**Integral (%)**	**$^2T_{Al}$**	**Integral (%)**	**T_{Al} total**	**$^3T_{Al}$**	**Integral (%)**	**Width (ppm)**
H8	57.9	36.6	54.4	42.6	79.2	0.4	20.8	1.8
H10	57.9	42.5	54.5	31.2	73.7	0.5	26.3	1.9
H12	57.9	31.3	54.4	44.6	75.9	0.4	24.1	2.1
H24	57.5	42.4	54.0	44.7	87.1	0.3	12.9	1.7
H48	60.9	51.1	56.6	16.5	67.5	3.0	32.5	1.4
H72	57.5	35.4	53.8	53.9	89.3	0.3	10.7	1.5

$^1T_{Al}$, $^2T_{Al}$ and $^3T_{Al}$ is the concentration of FAl and EFAL peaks in ppm

T_{Al} is total % integral of $^1T_{Al}$ and $^2T_{Al}$

Supplementary Table S3. Chemical shift, Si/Al ratio, intensity and area of deconvoluted Q^3 and Q^4 peak.

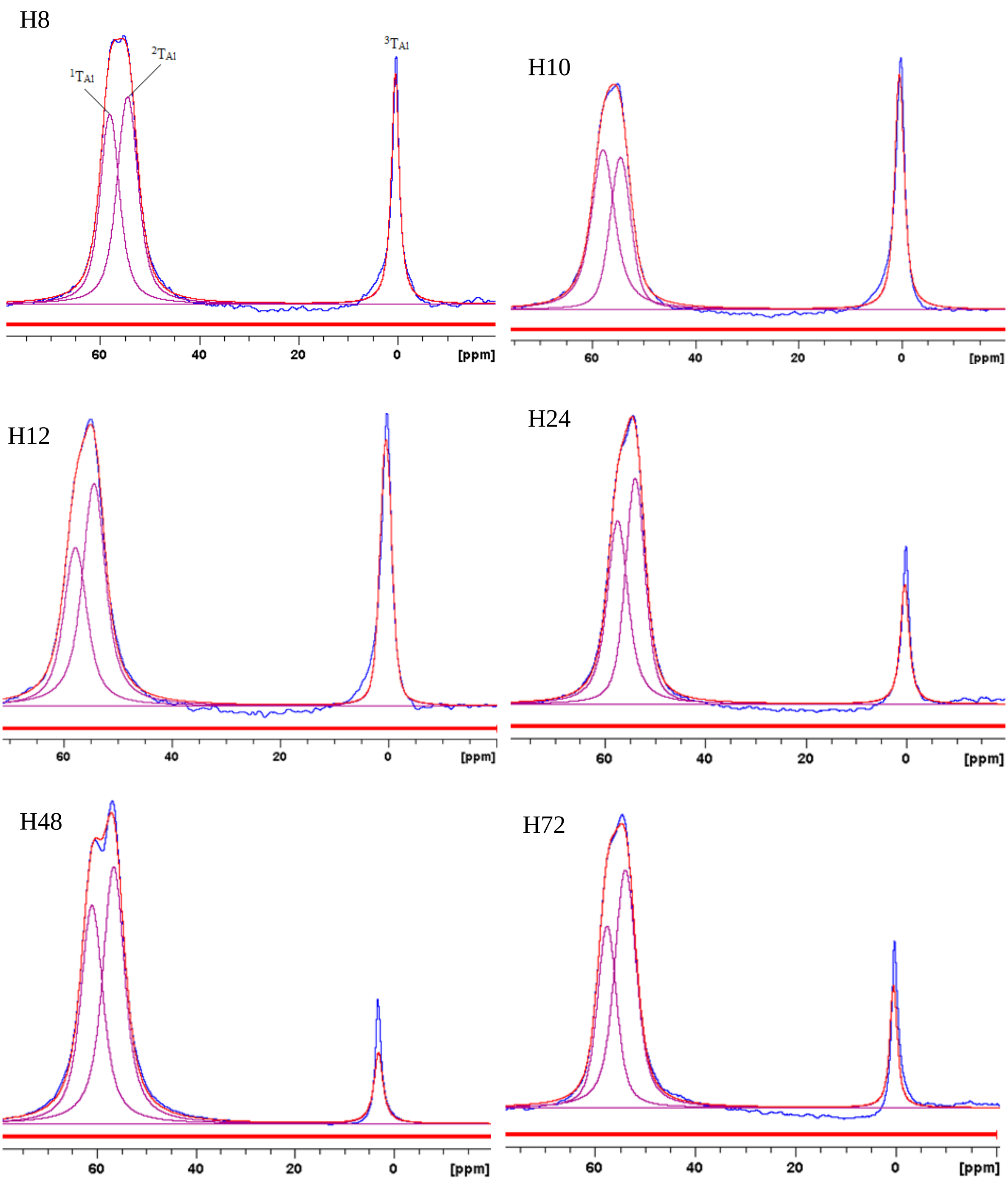
Samples	Q^3 Si(1Al)		Q^4 Si(0Al)		NMR Si/Al ratio	EDS Si/Al ratio
	δ_{decon} (ppm)	Area %	δ_{decon} (ppm)	Area %		
H8	-102.9	23.7	-111.0	41.1	29.7	30
H10	-104.4	25.1	-111.1	43.4	26.8	25
H12	-104.2	22.8	-111.3	45.5	30.0	25
H24	-103.3	24.6	-110.7	45.8	24.8	24
H48	-103.3	27.1	-110.9	38.9	23.7	26
H72	-103.1	26.3	-110.8	42.9	22.9	23

δ_{decon} determined from NMR chemical shifts.

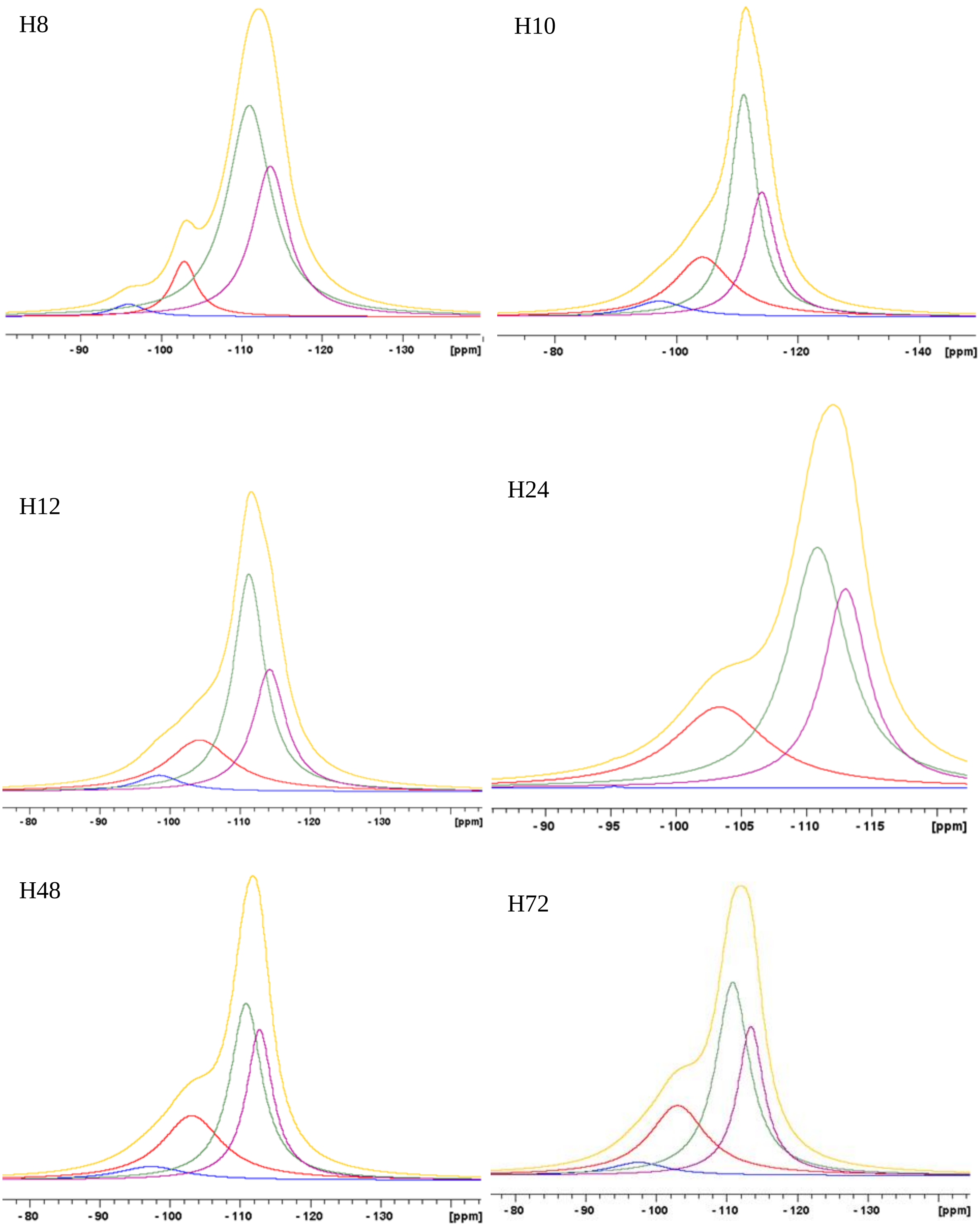
The derived framework Si/Al ratio was calculated according to equation below (Holzinger et al., 2018).

$$\frac{Si}{Al}(^{29}\text{Si}) = \frac{2.I[Q^2\{2Al\}]+3.I[Q^3\{1Al\}]+4.I[Q^4\{0Al\}]}{I[Q^3\{1Al\}]}$$

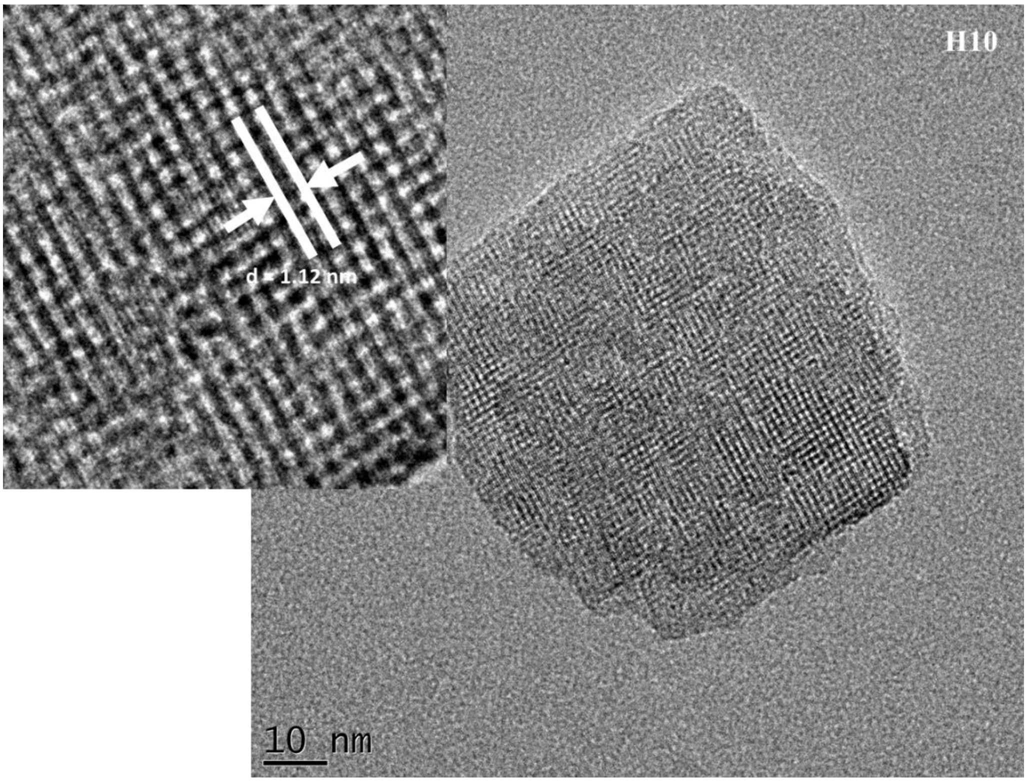
where I is the peak intensity of Q^2, Q^3 and Q^4 resonances identified by deconvolution of the ^{29}Si MAS NMR spectra.

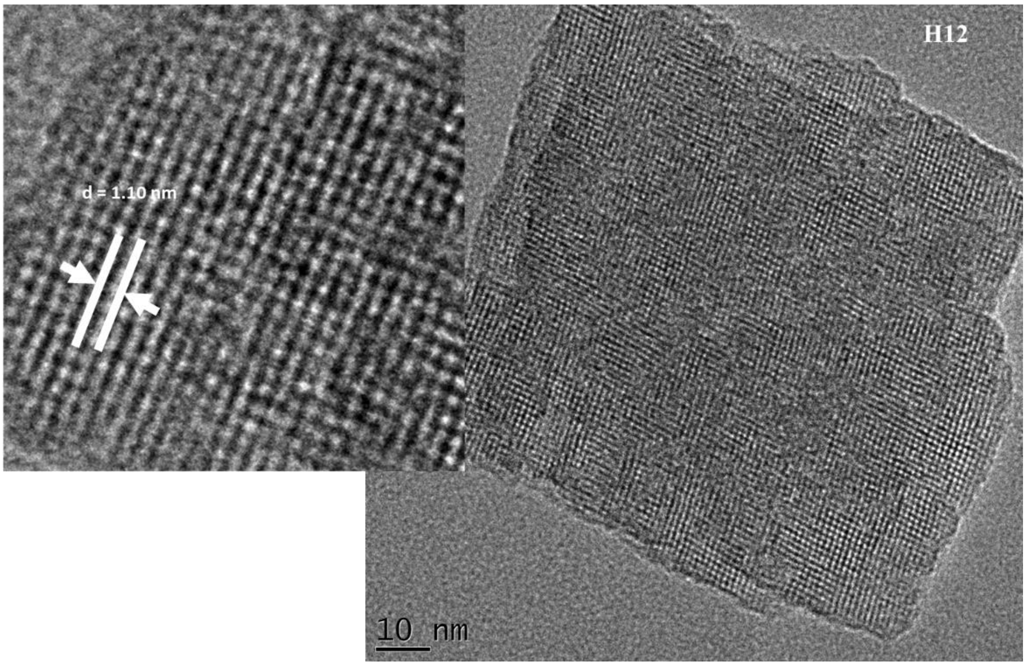


Supplementary Figure S2. Deconvoluted ^{27}Al MAS NMR spectra of HBEA zeolites.



Supplementary Figure S3. Deconvoluted ^{29}Si MAS NMR spectra of HBEA zeolites.


H10
d = 1.12 nm
10 nm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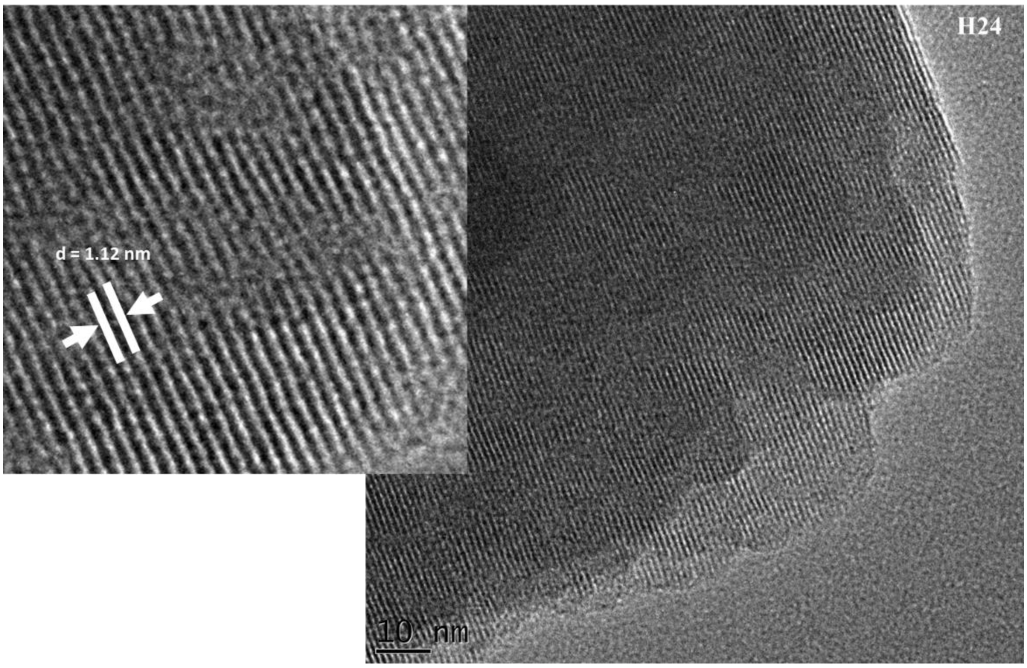

Supplementary Figure S4. TEM images of as-synthesised HBEA zeolites indicating the d-space.

References:

Holzinger, Julian, Pablo Beato, Lars Fahl Lundegaard, and Jørgen Skibsted. "Distribution of aluminum over the tetrahedral sites in ZSM-5 zeolites and their evolution after steam treatment." The Journal of Physical Chemistry C 122, no. 27 (2018): 15595-15613.