

## Supplementary Information

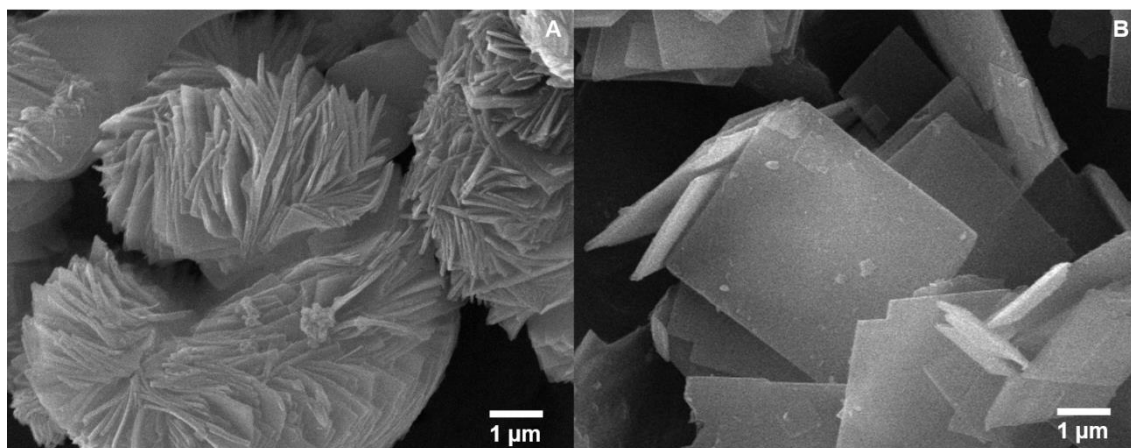
### Vapor adsorption experiments as a characterization tool for layered catalysts

Guilherme P. Campos<sup>a</sup>, Gabriel B. Báfero<sup>a</sup>, Heloise O. Pastore<sup>\*a</sup>

<sup>a</sup> Institute of Chemistry, University of Campinas, Monteiro Lobato St. 270, 13084-971, Campinas, Sao Paulo, Brazil.

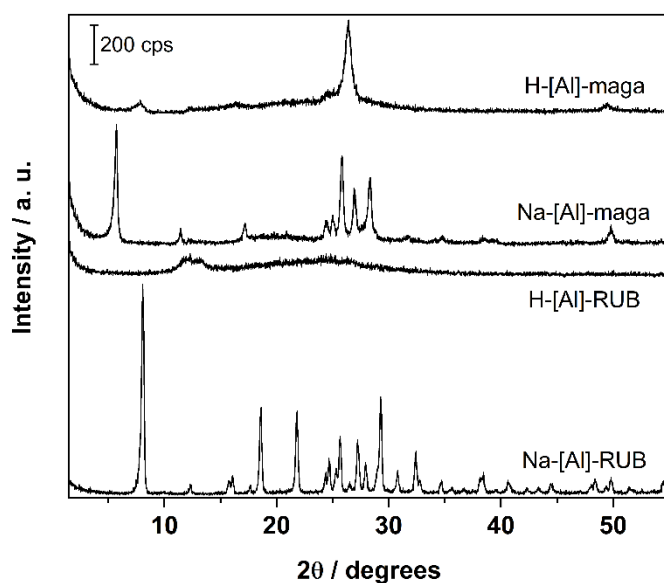
Corresponding e-mail: gpmmm@unicamp.br

#### 1. Scanning electron micrography of Na-[Al]-magadiite and Na-[Al]-RUB-18.



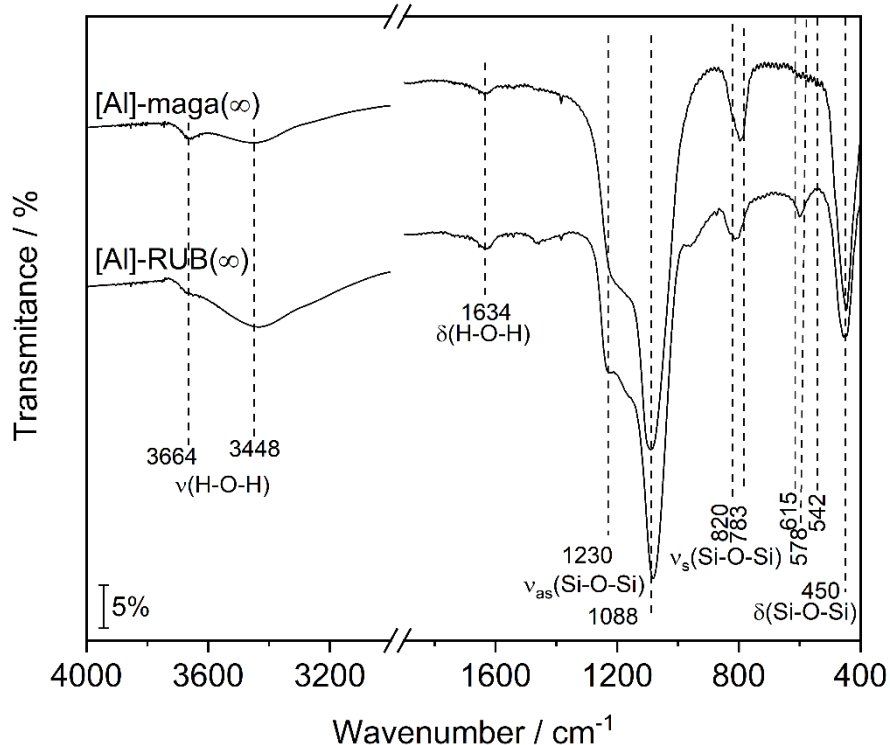
**Fig. S1.** Scanning electron micrography of Na-[Al]-magadiite (A) and Na-[Al]-RUB-18 (B) samples.

## 2. X ray diffraction of sodium and protonic magadiite and RUB-18.



**Fig. S2.** X ray diffraction of the synthesized materials (Na-[Al]-maga and Na-[Al]-RUB) and after ammonia thermodecomposition (H-[Al]-maga and H-[Al]-RUB).

## 3. Fourier Transform Infrared (FTIR) of protonic magadiite and RUB-18.



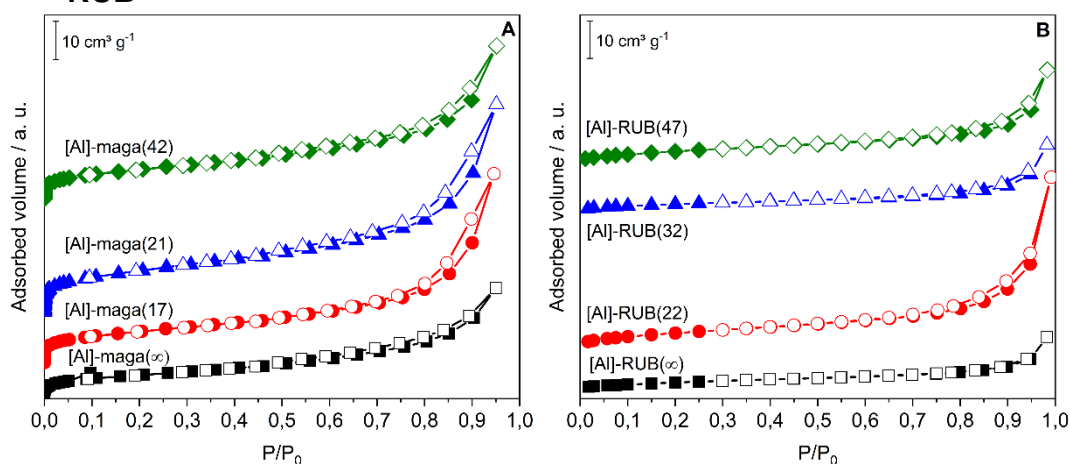
**Fig. S3.** Examples of FTIR spectrum of protonic materials after the thermodecomposition of ammonia.

**Table S1.** Assignment of FTIR bands of the layered materials<sup>1</sup>

Wavenumber [cm <sup>-1</sup> ]	Assignment	Observation
3664; 3448	$\nu(\text{H-O-H})$	water molecules and/or silanols groups
1634	$\delta(\text{H-O-H})$	water molecules vibration
1230; 1088	$\nu_{\text{as}}(\text{Si-O-Si})$	internal vibration of 5 member rings present in the solids structure
820; 783	$\nu_{\text{s}}(\text{Si-O-Si})$	vibrations of TO4 groups in the structure
615; 578		double ring vibration present in the solids structure
450; 443	$\delta(\text{Si-O-Si})$	deformation of Si-O-Si groups

1: Y. Huang, Z. Jiang, W. Schwieger, *Chemistry of materials*, 1999, **11**, 1210-1217.

#### 4. Surface and porosity characterization of [Al]-magadiites and [Al]-RUB

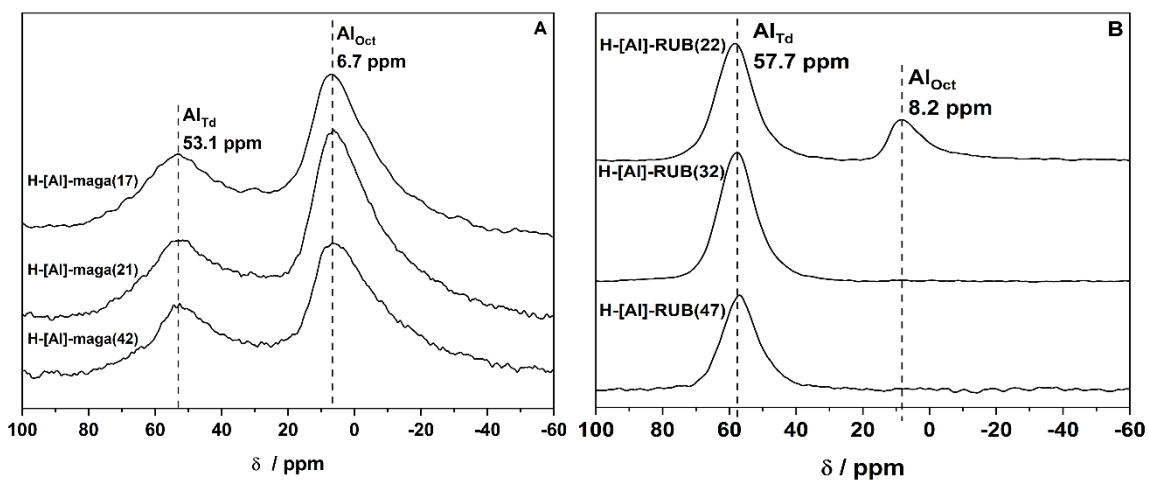


**Fig. S4.** Physisorption of N<sub>2</sub> in H-[Al]-magadiites (A) and Ar in H-[Al]-RUB-18 samples (B).

**Table S2.** Surface area and total pore volume data obtained by N<sub>2</sub> and Ar physisorption

Protonic solid	Si/Al	A <sub>BET</sub> / m <sup>2</sup> g <sup>-1</sup>	Total por volume / cm <sup>3</sup> g <sup>-1</sup>
[Al]-maga	∞	21.8	0.04
	17	24.7	0.08
	21	40.9	0.09
	42	29.4	0.06
[Al]-RUB	∞	6.7	0.02
	22	12.5	0.06
	32	7.3	0.02
	47	10.6	0.03

## 5. Solid state nuclear magnetic resonance of $^{27}\text{Al}$ nuclei



**Fig. S5.** Solid state NMR of  $^{27}\text{Al}$  nuclei of (A) for H-[Al]-magadiite samples and (B) H-[Al]-RUB samples.

**Table S3.** Ethanol adsorption and materials data.

Sample	$V_{\text{ads}}$ [cm <sup>3</sup> g <sup>-1</sup> ]	$n_{\text{et}}$ [mol EtOH/g]	Al <sub>Td</sub>	Al <sub>Oct</sub>	$n_{\text{Al}}$ [moles of Al/g]	$V_{\text{ads}}/n_{\text{Al}}$ [L of EtOH/moles of Al]	$n_{\text{et}}/n_{\text{Al}}$ [moles of EtOH/moles of Al]
Na-maga( $\infty$ )	2.5	4.3E-2	-	-	-	-	-
Na-[Al]-maga(17)	3.3	5.6E-2	-	-	8.7E-04	3.8	64.7
Na-[Al]-maga(21)	3.3	5.6E-2	-	-	7.1E-04	4.7	79.0
Na-[Al]-maga(42)	2.8	4.8E-2	-	-	3.7E-04	7.7	131.5
H-maga( $\infty$ )	6.4	1.1E-1	-	-	-	-	-
H-[Al]-maga(17)	3.9	6.8E-2	28%	72%	8.7E-04	4.5	78.6
H-[Al]-maga(21)	5.7	9.8E-2	31%	69%	7.1E-04	8.0	138.2
H-[Al]-maga(42)	6.1	1.0E-1	36%	64%	3.7E-04	16.7	274.0
H-RUB( $\infty$ )	0.7	1.2E-2	-	-	-	-	-
H-[Al]-RUB(22)	4.0	6.9E-2	76%	24%	4.5E-04	9.0	154.7
H-[Al]-RUB(32)	4.7	8.1E-2	100%	-	4.3E-04	10.9	187.5
H-[Al]-RUB(47)	5.0	8.7E-2	100%	-	3.0E-04	16.7	291.0

$n_{\text{et}}$ : moles of ethanol adsorbed per gram of material.

$n_{\text{Al}}$ : moles of aluminum per gram of material determined by X ray fluorescence.

$V_{\text{ads}}/n_{\text{Al}}$ : volume adsorbed of ethanol adsorbed per moles of aluminum.

$n_{\text{et}}/n_{\text{Al}}$ : moles of ethanol per moles of aluminum.

**Table S4.** Pentane adsorption and materials data.

Sample	$V_{ads}$ [cm <sup>3</sup> g <sup>-1</sup> ]	$n_p$ [mol pentane/g]	Al <sub>Td</sub>	Al <sub>Oct</sub>	$n_{Al}$ [moles of pentane/g]	$V_{ads}/n_{Al}$ [L of pentane/moles of Al]	$n_p/n_{Al}$ [moles of /moles of Al]
H-maga( $\infty$ )	3.0	2.6E-2	-	-	-	-	-
H-[Al]-maga(42)	3.9	3.4E-2	36%	64%	3.7E-04	10.7	93.2
H-[Al]-RUB( $\infty$ )	1.0	8.9E-3	-	-	-	-	-
H-[Al]-RUB(47)	5.1	4.4E-2	100%		3.0E-04	17.1	148.7

$n_p$ : moles of pentane adsorbed per gram of material.

$n_{Al}$ : moles of aluminum per gram of material determined by X ray fluorescence.

$V_{ads}/n_{Al}$ : volume adsorbed of pentane adsorbed per moles of aluminum.

$n_p/n_{Al}$ : moles of pentane per moles of aluminum.