Electronic Supplementary Material (ESI) for RSC Advances

Electronic Supplementary Information

Ladder-like aminopropylsilsesquioxane. Nice alternative for controlled drug delivery

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Characterization methods

Elemental analyses were carried out by the "Service Central de Micro-Analyse du CNRS, Vernaison, France".

The nitrogen adsorption isotherms were measured at liquid temperature (77 K) using a Micromeritics Tristar 3000 analyser. Before the measurements, the samples were out gassed under vacuum for 12 h at 100 °C. The specific surface areas were calculated by the Brunauer-Emmett-Teller (BET) method.

Scanning Electron Microscopy (SEM) images were obtained with a Hitachi S2600N microscope.

Powder XRD experiments were carried out with a high resolution Bonse–Hart camera with two germanium channel cuts for very small θ values. The wavelength used was 1.542 Å (Cu-Kα radiation).

TGA profiles were recorded between 25 and 700 °C with a slope of 5 °C/min under air with a Netzsch STA 409 PC apparatus using ca. 10 mg samples in Pt pans

Solid state NMR spectra for ^{29}Si were recorded on a Varian VNMRS 300 Solid spectrometer at a magnetic field strength of 7.05 T. A 7.5 mm MAS probe was used with a spinning rate of 5 kHz. Single pulse experiments with a continuous wave ^{1}H decoupling were used for ^{29}Si NMR, with 2 μs π/2 pulse duration and a recycle delay of 60 s. A recycle delay of 200 s was found necessary to allow full ^{29}Si relaxation although this did not lead to any change in the relative ratio of the individual components on spectral decomposition. Thus, data with recycle delay of 60 s can be considered as quantitative.

The *in vitro* release tests of ibuprofen were performed in various physiological simulated fluids at pH 1.3 (HCl/NaCl) and pH= 5.0 to 8.0 (KH_2PO_4/Na_2HPO_4), on roughly 0.100 g of material weighted with precision. Tests were done for 24 hours min at 37°C, in a USP apparatus 4 (cell powder CE1, Sotax™), in closed loop configuration with a flow fixed at 9.0 ml/min. Amounts of drug released were determined by HPLC (LC-2010Aht, Shimadzu) on a reversed C18 column, with UV-visible adsorption detector (λ = 260 nm).

Experimental Section

a. Materials

Aminopropyltrimethoxysilane (APTMS) and ibuprofen sodium were obtained from Aldrich and used as received without further purification.

b. Preparation of **IBU**

Ibuprofen (**IBU**) was prepared by treatment of ibuprofen sodium (12.0 g) with 100 mL of 2M HCl solution and extracted with diethyl ether (3x50 mL). After drying of organic layers over $MgSO_4$, the solvent was removed under vacuum to give 8.1 g of ibuprofen as white solid.

c. Preparation of the **M-IBU**

0.94 g (9 mmoles) of material **M** was added to a solution of ibuprofen (2.23 g, 10.8 mmoles) in THF (25 mL). The resulting suspension was stirred under reflux for 24h and the beige solid was filtered off and washed 3 times with 10 mL of THF. After drying under vacuum at 70 °C for 5h, 2.10 g (78 %) of **M-IBU** were obtained.

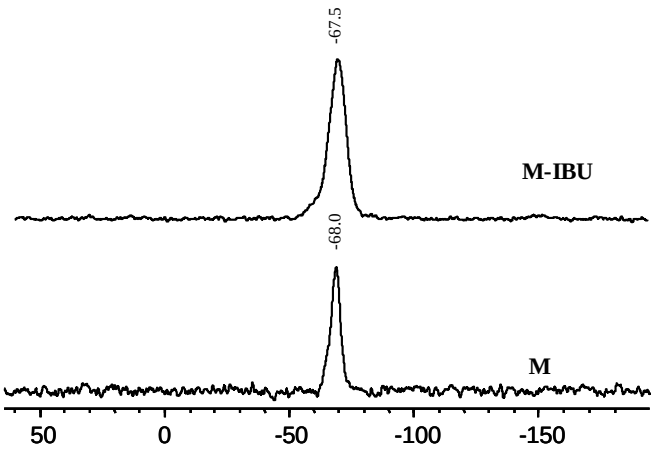

Fig. S1. CP-MAS ^{29}Si NMR spectra of **M** and **M-IBU**.

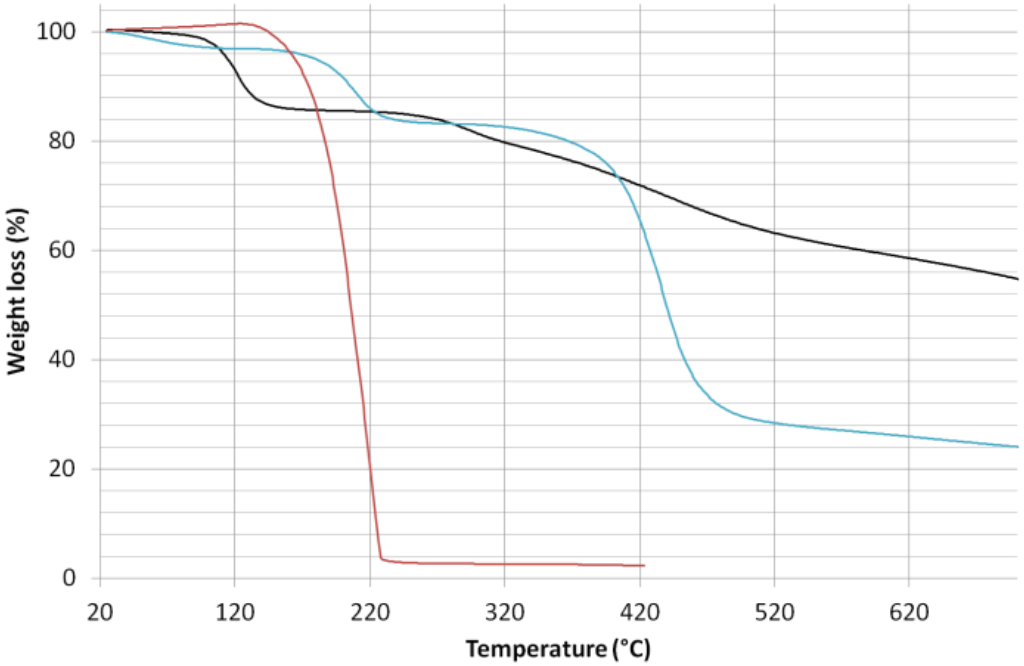

Fig. S2. TGA curves of **M** (black), **IBU** (red) and **M-IBU** (bleu).

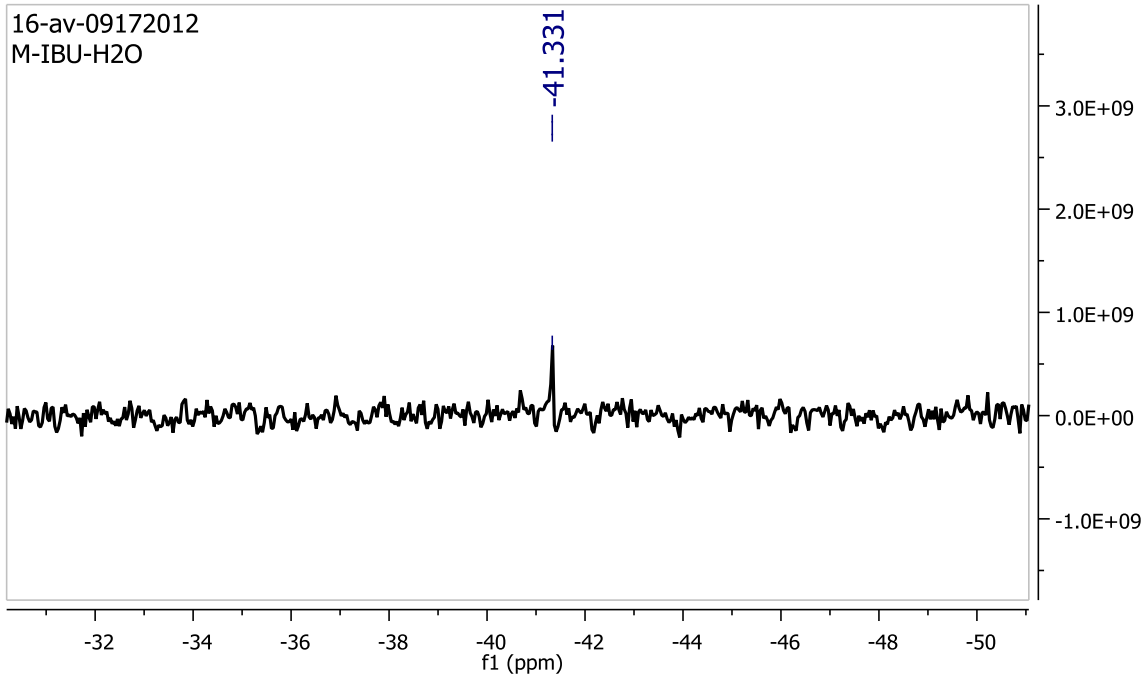

Fig. S3. ^{29}Si NMR spectrum of **M-IBU** after 48h in water at pH = 8.