Electronic Supplementary Material (ESI) for Chemical Communications

Encapsulated Recyclable Porous Materials: An Effective Moisture-Triggered Fragrance Release

Bisera Efremovska,[‡] Haohan Wu,[‡] John Vaughn,[‡] David H. Olson, Jairajh Mattai, Claudio Ortiz, Allen Puchalski, Jing Li, and Long Pan**

SUPPORTING INFORMATION

Table of Contents

General Procedures

Powder X-ray diffraction

Powder X-ray diffraction experiment was conducted using a D/M-2200T automated system (Ultima+, Rigaku) with Cu Kα radiation (λ = 1.5406 Å). The PXRD patterns were collected between 3° and 50° (2θ) at a scan rate of 5 deg/min (40 kV, 40 mA).

Thermogravimetric Analysis (TGA).

The activation temperature of the fragrance bubbler was 18 ºC. When loading the RPM with fragrance, the framework was first dried in an oven vacuum at 135 ºC overnight. Once the RPM was dried and all moisture eradicated, excess ethyl butyrate was added over a constant flow of nitrogen gas for a period of two hours at a temperature of 30 ºC. Once the sample was loaded with the fragrance, it was placed in the TGA and adsorption uptake was measured every thirty minutes. This procedure was repeated for both fragrances.

Using RPM_3-Zn, the rate of adsorption was measured at 30 ºC and variable pressure. The parameters for this experiment were as follows: activation condition is 120 minutes at 120 ºC, 200 minutes for each adsorption and desorprtion – recording weight every minute. The pressures were as follows: 3.2 torr, 5.4 torr, 8.2 torr, and 11.1 torr.

Headspace GC-MS.

Samples were prepared for GC-MS analysis such that there was 3.5 mg of fragrance compound (either ethyl butyrate or D-limonene). The sample cap was placed on top of the vial, crimped shut, and placed in a water bath at 37 ºC. A 1mL aliquot of headspace was taken, by inserting the syringe through the septum to a depth of approximately 5cm, measured from the top of the vial. The instrument used is an Agilent GC/MS Model 6890, 5973 Network Mass Selective Detector with Gerstel Multipurpose Headspace sampler MPS2. The method developed for the Gerstel Headspace is as follows: 10 minute injection time, 250 rpm agitation speed, 37 ºC syringe temperature, 500 uL/s injection speed for a total of 20 minutes cycle time. For the GC/MS instrument the method developed is as follows: 1.00 mL/min column flow, 7.56 psi pressure, Helium gas, run time total of 20 minutes. The column used is Agilent 190091S-433, 350 ºC max, HP-5MS 0.25mm x 30.0m x 0.25um, capillary 30.0m x 250um x 0.25um nominal. The ramp was set at 12 ºC/minute starting at 45 ºC until 250 ºC, holding for 1.92 minutes with 0.50 minute solvent delay. Ethyl butyrate was assigned to the signal at ~3.36 min, and d-limonene was assigned as ~6.67 min. Abundance was determined by peak height. Experimental data are listed in table S1, S2 and S3.

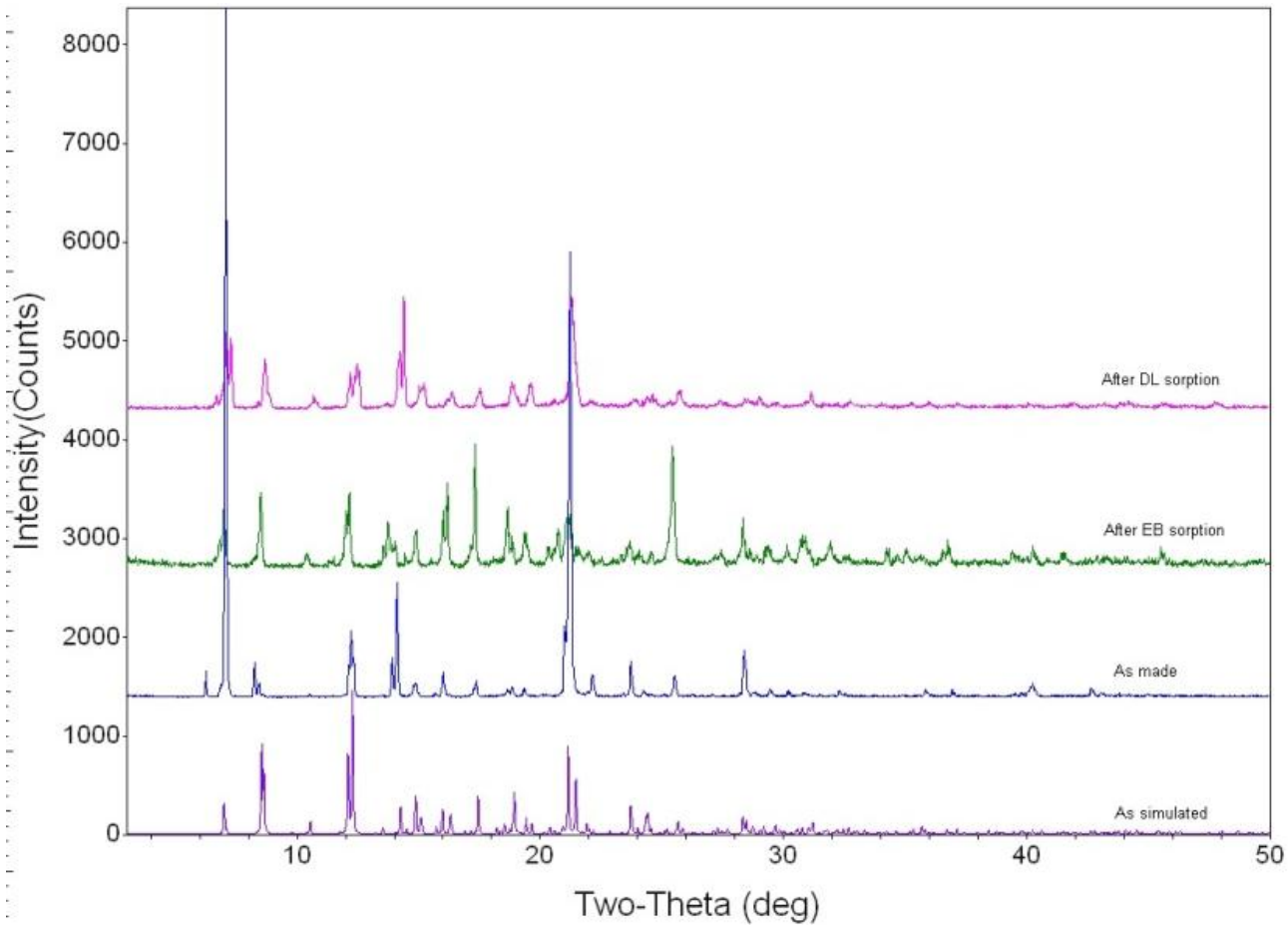

Figure S1a. The PXRD patterns of RPM1-Zn before and after adsorption experiments. Color scheme: purple, simulated; blue, as made; green, after EB sorption experiment; pink, after DL sorption experiment.

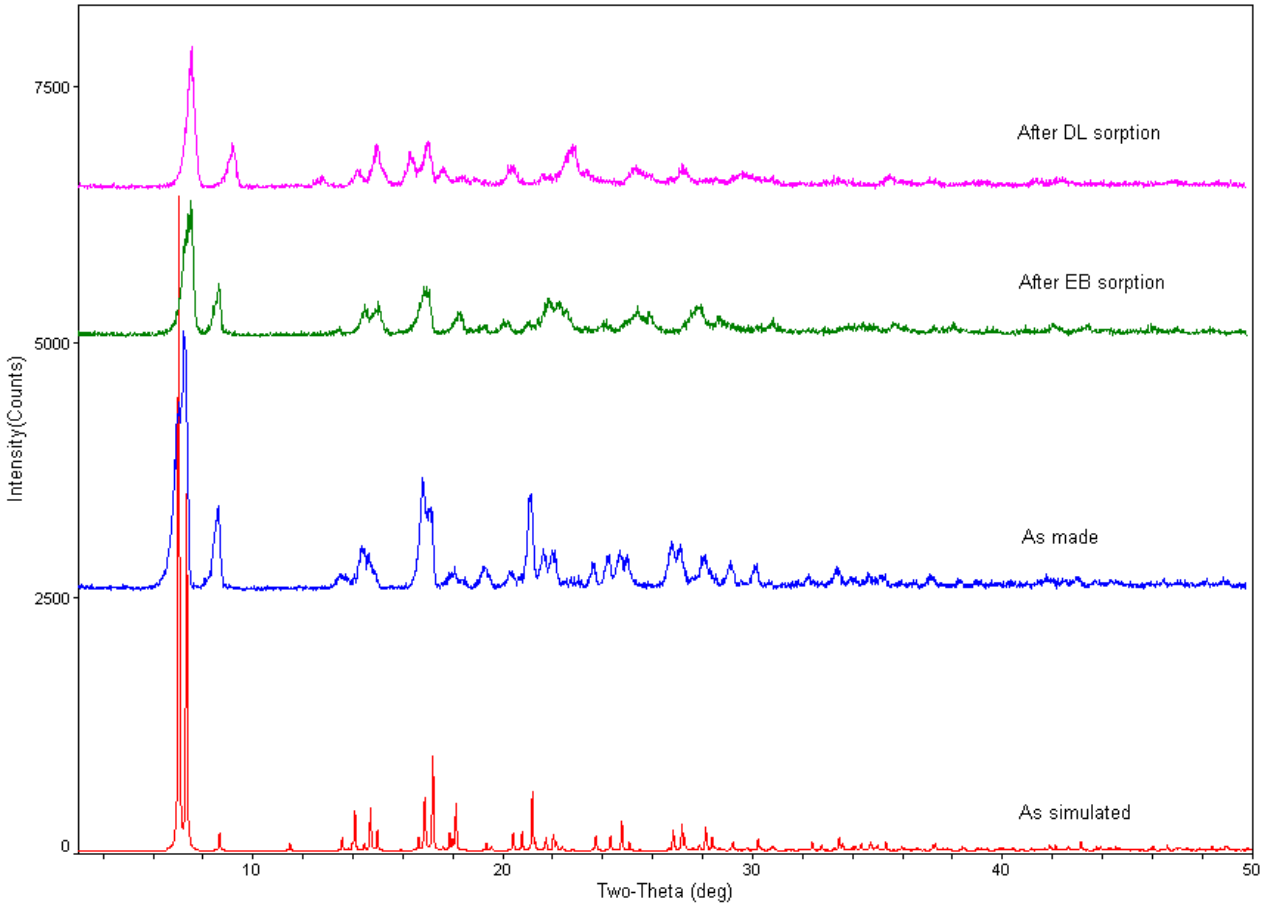

Figure S1b. The PXRD patterns of RPM3-Zn before and after adsorption experiments. Color scheme: red, simulated; blue, as made; green, after EB sorption experiment; pink, after DL sorption experiment.

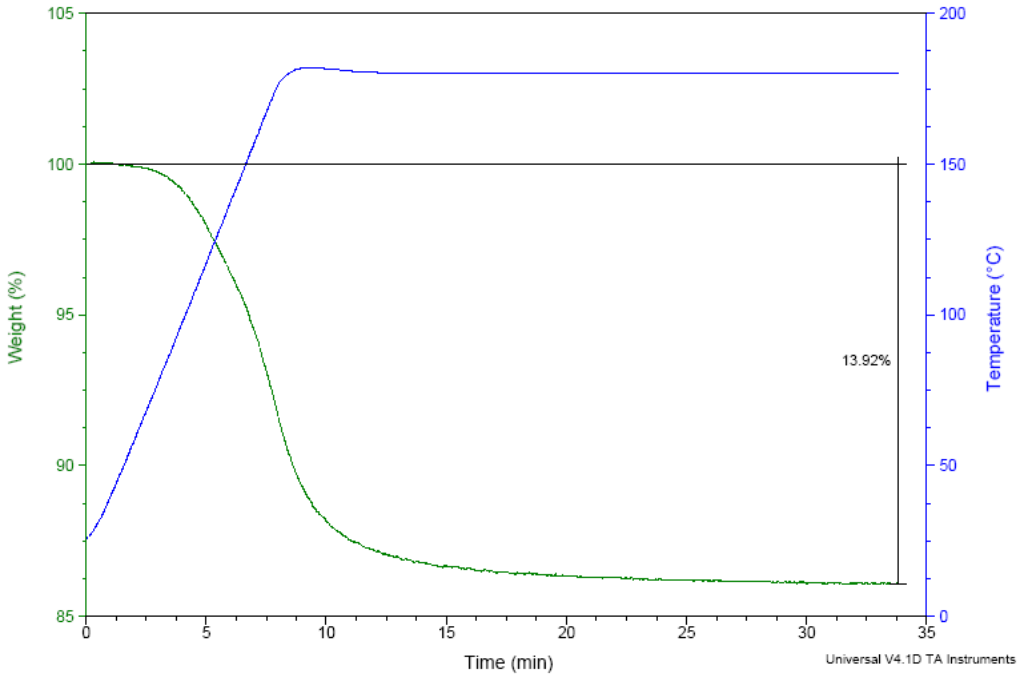

Figure S2a. The TG profile of EB desorption from RPM3-Zn.

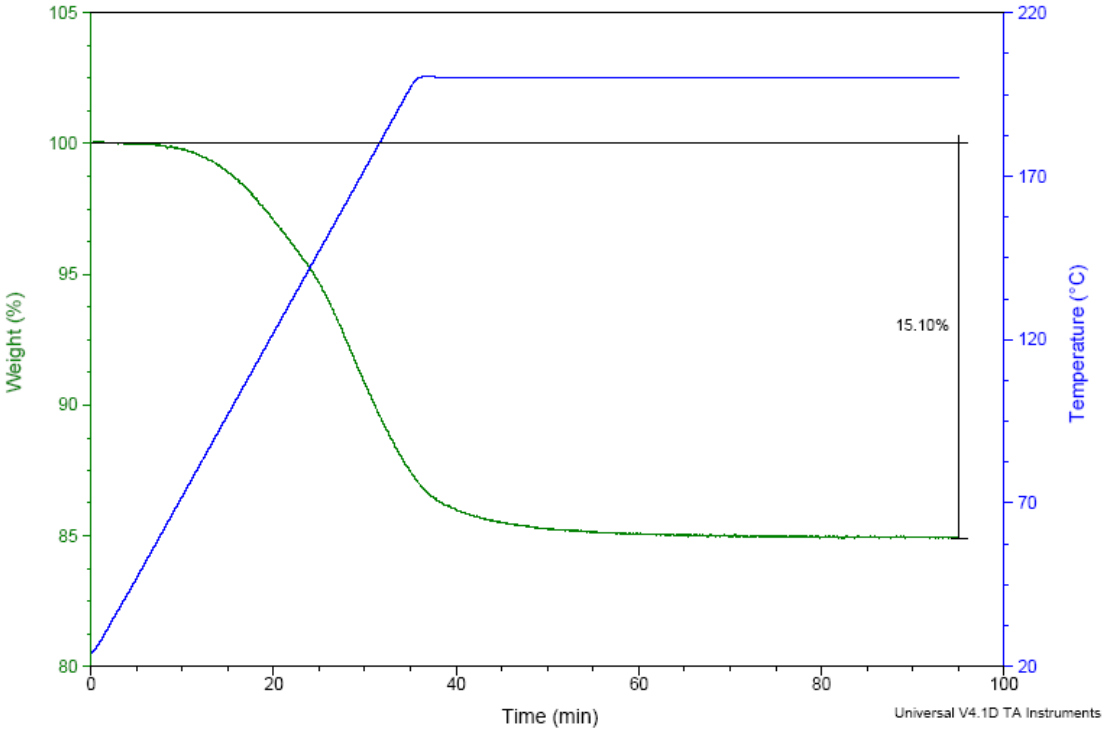

Figure S2b. The TG profile of DL desorption from RPM3-Zn.

Table S1. GC-MS Analysis of D-Limonene

Time [Hr]	Peak Height [AU]
0	0
0.4833	3379618
1.01667	3904776
1.4667	3941621
2.05	3947061

Table S2. GC-MS Analysis of RPM-Cap@DL

Time [Hr]	Peak Height [AU]
0	1583680
0.7	4696887
1.3833	5312405
2.1667	5073747
2.6	5162841
3.6833	5347841
4.15	5530218

Table S3. GC-MS Analysis of Modified Starch + DL

Time [Hr]	Peak Height [AU]
0	4723039
0.58333	5104934
1.216667	4850220
1.7333	4958113
2.45	5137576
2.9833	5214892
3.7667	5133303
3.98333	4938147