Supporting Information

Synthesis of ZIF-11 Crystals by Microwave Heating

Jolie Lucero [a], Taylor J. Self [a], Moises A. Carreon *

Chemical and Biological Engineering Department, Colorado School of Mines, Golden, CO 80401

[a] These authors contributed equally to this work

* Corresponding author: mcarreon@mines.edu

Experimental

Materials

Optima ® Methanol (Fisher Chemical), and ACS grade toluene (LabChem) were used as solvents in the synthesis of ZIF-11. Benzimidazole, 98% (ACROS Organics), zinc acetate dihydrate, 98% (Sigma Aldrich), and ammonium hydroxide, 28-30% (Sigma Aldrich) were used for ZIF-11 formation. All chemicals were used as received.

Additional Experimentation

To validate the effect of the microwave, an additional experiment was performed. The procedure detailed in the manuscript was followed except for the final mixing step. Once the two homogenous solutions were prepared, they were mixed together and stirred on a stir plate at room temperature for only 2 minutes. Then the mixture was added to the microwave under the same conditions as before. 100°C was the holding temperature for 15 minutes, the microwave stirring feature was used.

Characterization

Powder X-Ray Diffraction. ZIF-11 crystals were analyzed using a Siemens Kristalloflex800, operated at 25 mA, 30 kV, Cu Kα radiation. Two theta ranges were from 2θ° = 2°- 40°.

Scanning Electron Microscopy. FE-SEM images were collected on a JEOL ISM-7000F with an accelerating voltage of 2.0 – 3.0 kV.

Surface Area Analysis. BET surface areas, and adsorption-desorption isotherms were collected from nitrogen isotherm data collected at 77 K, on a Micromeritics, ASAP 2020 porosity analyzer. Prior to BET analysis, samples were degassed at 180 °C, under vacuum for 6 hours.

Thermogravametric Analysis. Thermograms for the ZIF-11 was collected on a TA Instruments Q 150 operated on a temperature range from 23°C to 800°C at a ramp of 10°C/min.

Fourier Transform Infrared Spectroscopy. The FTIR spectrum was collected on a using 32 scans at 4 cm^{-1} resolution on a Thermo Scientific Nicolet iS50 FT-IR with a DTGS detector. ZIF-11 powder was incorporated into a KBr pellet.

Relative Crystallinity Calculation. To understand the relative crystallinity of each of the two samples, the area was taken under the leading peak, where 2θ°= 4.3. The relative crystallinity is calculated with the simulated pattern as 100% crystalline. The CIF file was taken from Sung Park et. al.[1]

$$\%\ Relative\ Crystallinity = \left(\frac{Area\ under\ 2\theta = 4.3}{Area\ under\ simulated\ 2\theta = 4.3}\right) x100\%$$

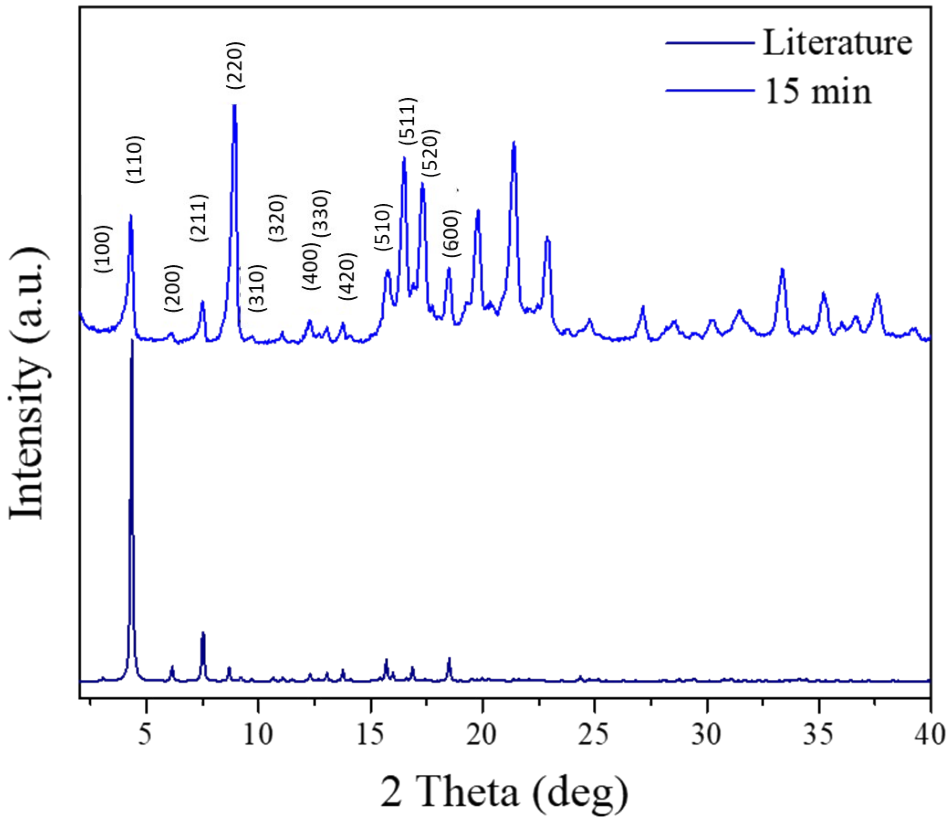

Figure S1. PXRD pattern of ZIF-11 synthesized by stirring both linker components for 2 minutes before addition to microwave.

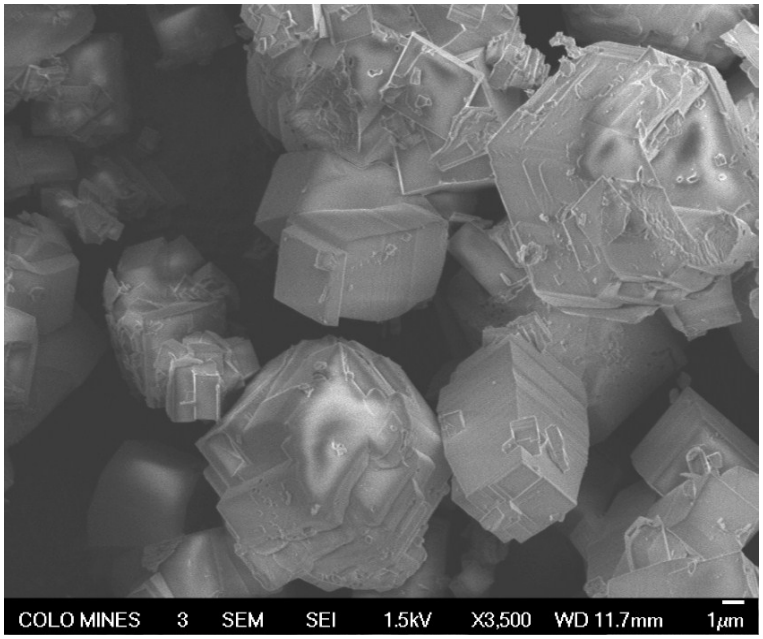

Figure S2. Representative SEM image of ZIF-11 with components stirred for 2 minutes before addition to microwave.

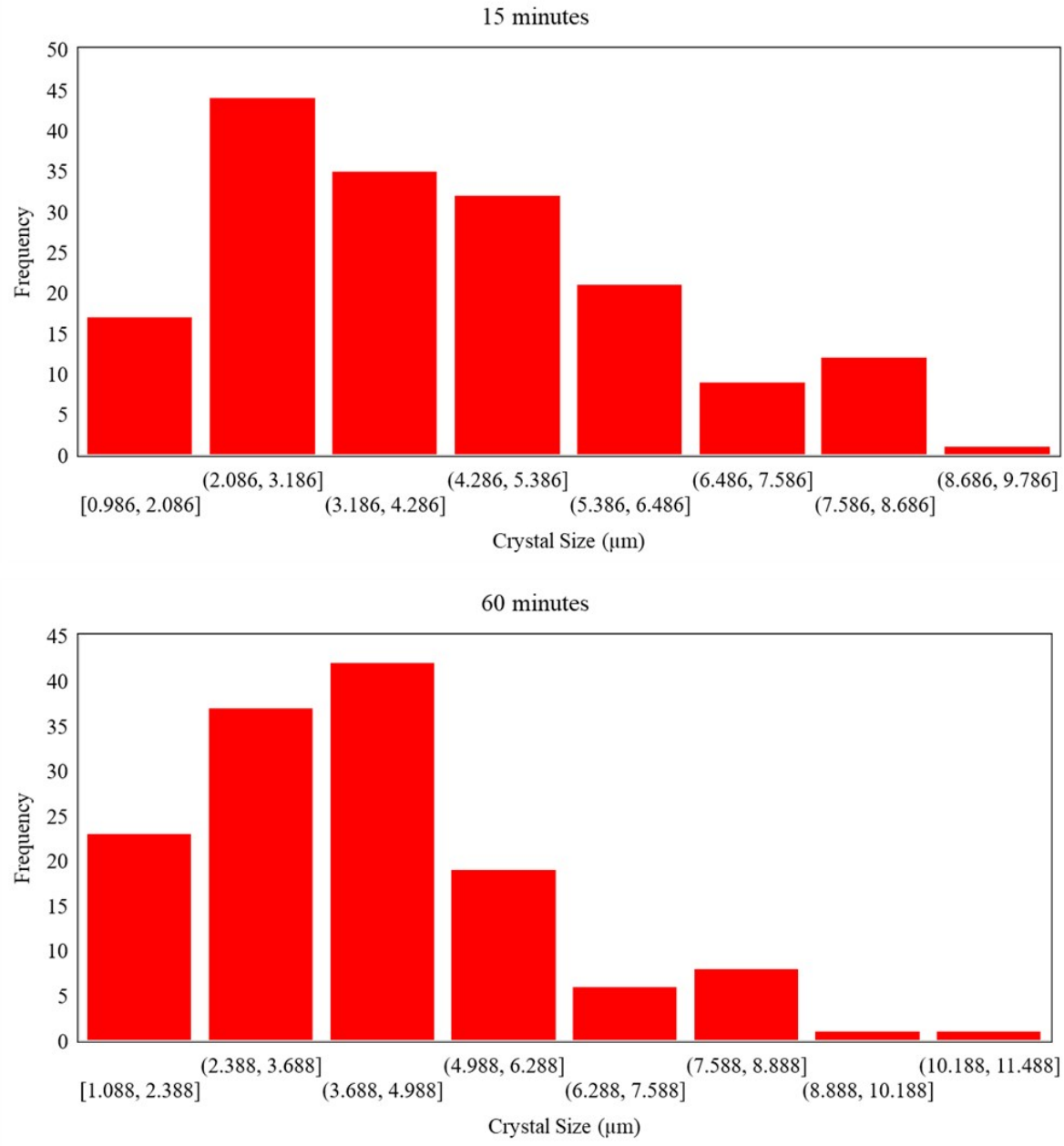

Figure S3. Size distribution histograms for crystals shown in Figure 2.

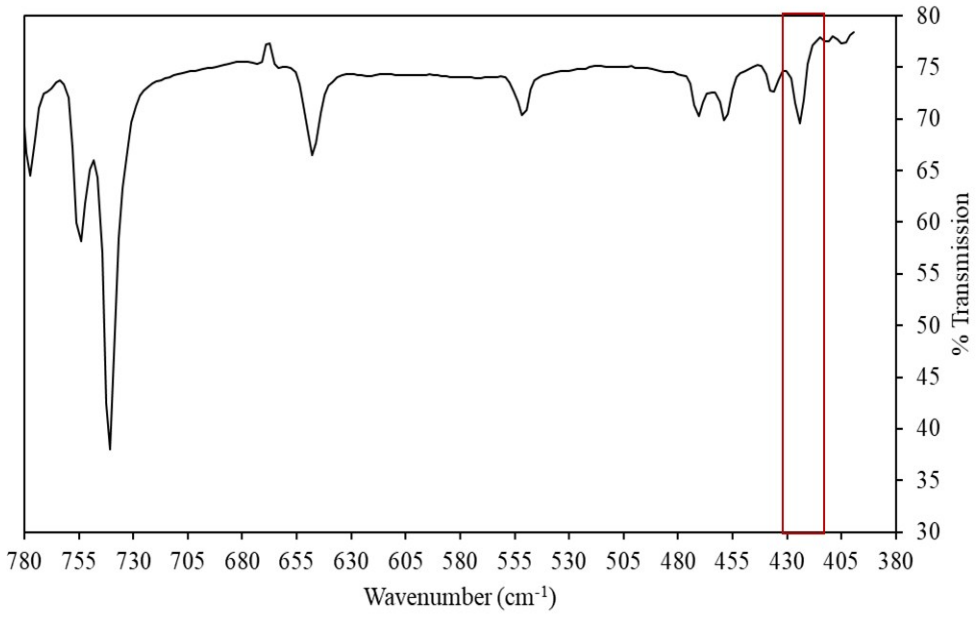

Figure S4. FTIR spectra of ZIF-11 synthesized via microwave at 15 minutes. Zn-N stretch at 427 cm^{-1} highlighted.

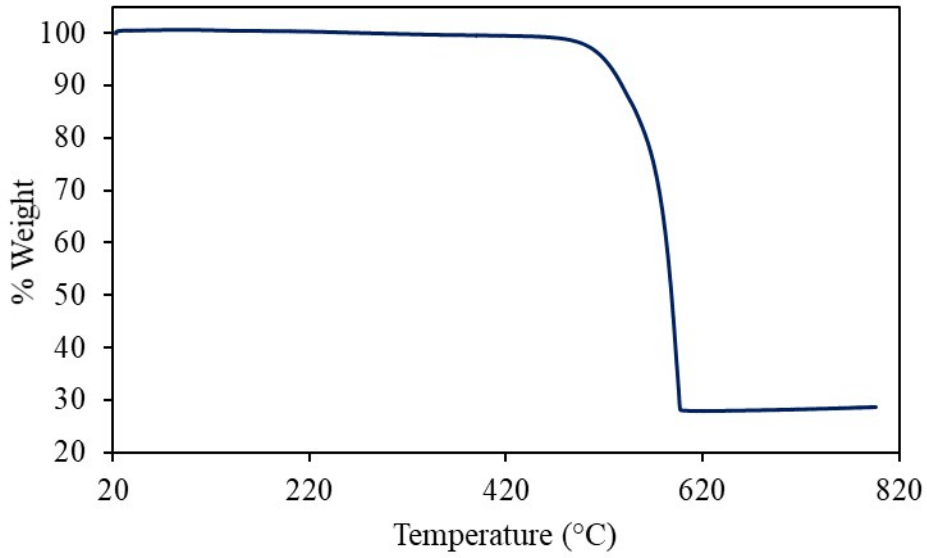

Figure S5. TGA of ZIF-11 synthesized via microwave for 15 min.

1. Park, K. S.; Ni, Z.; Côté, A. P.; Choi, J. Y.; Huang, R.; Uribe-Romo, F. J.; Chae, H. K.; O'Keeffe, M.; Yaghi, O. M., Exceptional chemical and thermal stability of zeolitic imidazolate frameworks. *Proceedings of the National Academy of Sciences* **2006,** *103* (27), 10186.