

## Electronic Supplementary Information (ESI†)

### Mechanochemical ZIF-9 Formation: In Situ Analysis and Photocatalytic Enhancement Evaluation

Noelia Rodríguez-Sánchez<sup>1,2</sup>, Carsten Prinz<sup>1</sup>, Ralf Bienert<sup>1</sup>, Menta Ballesteros<sup>2,3</sup>, A. Rabdel Ruiz Salvador<sup>2,4</sup>, Biswajit Bhattacharya<sup>1\*</sup>, Franziska Emmerling<sup>1,5\*</sup>

\*Corresponding authors: BB [biswajit.bhattacharya@bam.de](mailto:biswajit.bhattacharya@bam.de) and FE [franziska.emmerling@bam.de](mailto:franziska.emmerling@bam.de)

<sup>1</sup>BAM Federal Institute for Materials Research and Testing, Richard-Willstätter-Straße 11, 12489, Berlin, Germany.

<sup>2</sup>Center for Nanoscience and Sustainable Technologies (CNATS), Universidad Pablo de Olavide, Ctra. Utrera km. 1, 41013, Seville, Spain.

<sup>3</sup>Department of Molecular Biology and Biochemistry Engineering, Universidad Pablo de Olavide, Ctra. Utrera km. 1, 41013, Seville, Spain.

<sup>4</sup> Department of Physical, Chemical and Natural Systems, Universidad Pablo de Olavide, Ctra. Utrera km. 1, 41013, Seville, Spain.

<sup>5</sup>Department of Chemistry, Humboldt-Universität of Berlin, Brook-Taylor-Strasse 2, 12489 Berlin, Germany.

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## Study of adsorption kinetics models

Langmuir, Freundlich, and Temkin adsorption models were analyzed for mechanochemical ZIF-9 based on the dark period during photo-Fenton test. The calculations of these analyses were based on the equations [1-3] corresponding to Langmuir, Freundlich and Tempkin isotherms respectively:

$$\frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \frac{C_e}{q_{max}} \quad \text{Eq.1}$$

$$\ln q_e = \frac{1}{n} \ln C_e + \ln K_F \quad \text{Eq.2}$$

$$q_e = K_T \ln f + K_T \ln C_e \quad \text{Eq.3}$$

Where  $q_e$  is the equilibrium adsorption capacity ( $\text{mg g}^{-1}$ ),  $q_{\max}$  is the maximum adsorption capacity ( $\text{mg g}^{-1}$ ), and  $K_L$  and  $K_F$  are the Langmuir and Freundlich constants, respectively.  $K_T$  and  $f$  ( $\text{mg L}^{-1}$ ) are the Tempkin and binding constants, respectively.

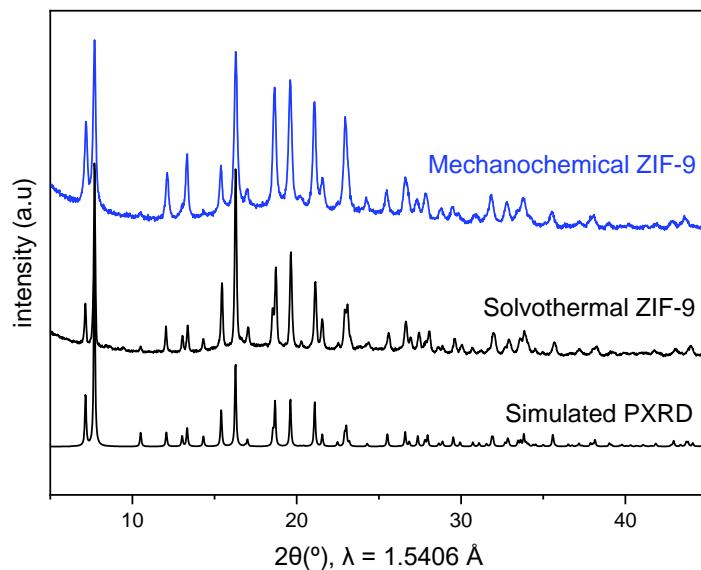


Fig. S.1. X-Ray Diffraction Patterns for solvothermal synthesized ZIF-9 (black) and mechanochemical synthesized ZIF-9 (blue).

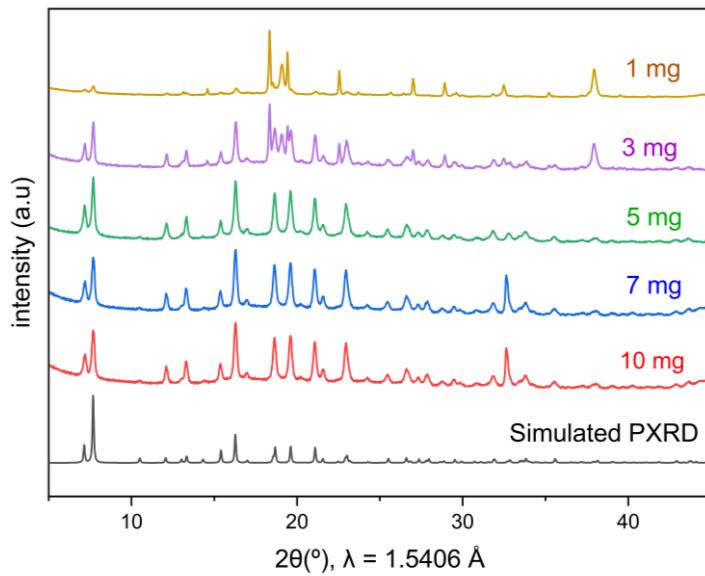


Fig.S.2. Evaluation of the concentration of  $\text{NH}_4\text{Cl}$  in the synthesis of mechanochemical ZIF-9.

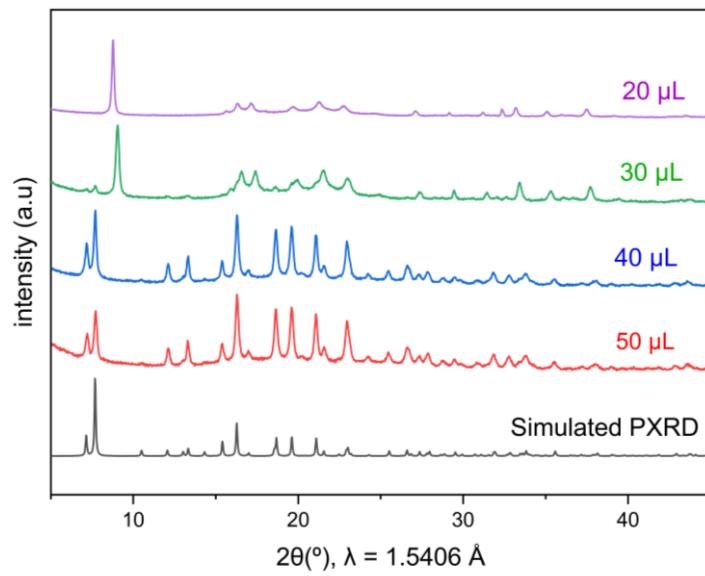


Fig. S.3. Evaluation of the concentration of DMF in the synthesis of mechanochemical ZIF-9.

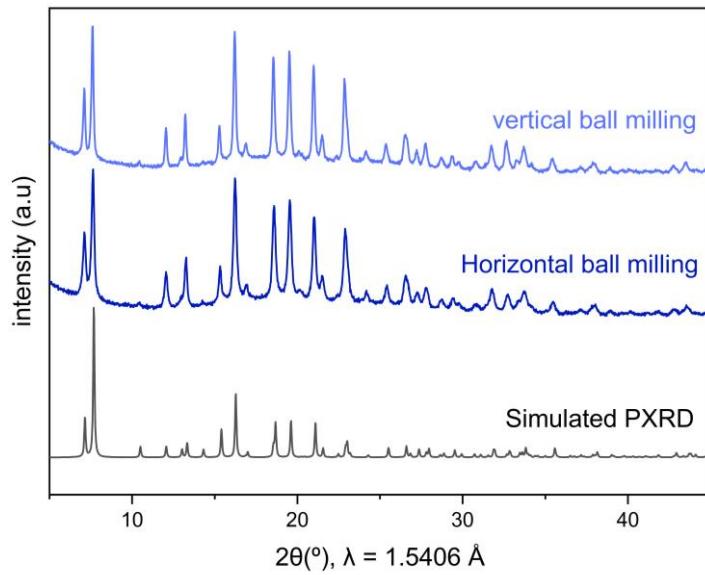


Fig. S.4. X-Ray diffraction patterns for mechanochemical ZIF-9 using a shaker mill with a vertical movement (light blue) and with horizontal movement (dark blue).

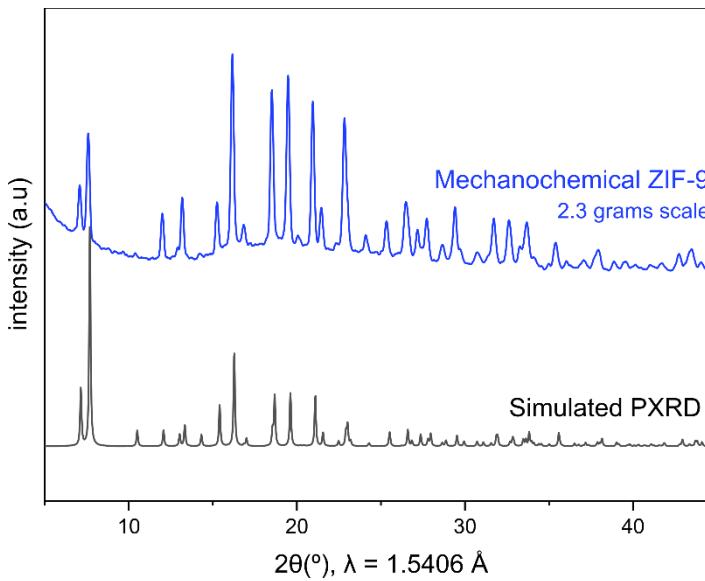


Fig. S.5. X-Ray diffraction patterns for 2.3 grams scale of mechanochemical ZIF-9.

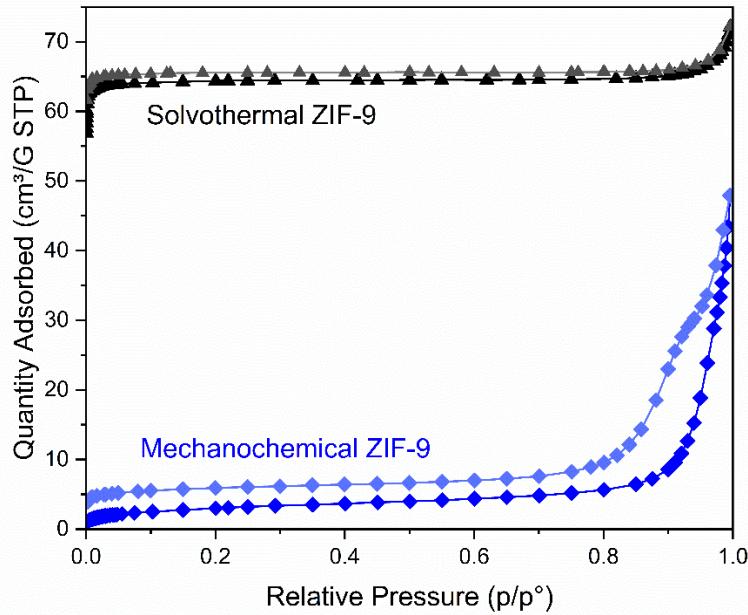


Fig.S.6. Adsorption isotherms for solvothermal synthetized ZIF-9 (black) and mechanochemical synthetized ZIF-9 (blue).

Table S.1. Specific surface area (BET) analysis of solvothermal and mechanochemical ZIF-9.

| Material              | Specific surface area (BET)<br>[m²/g] | Uncertainty (BET)<br>[m²/g] | C      | Cor. Coeff. | Measuring [p/p₀]  |
|-----------------------|---------------------------------------|-----------------------------|--------|-------------|-------------------|
| Solvothermal ZIF-9    | 274.06                                | $\pm 0.0652$                | 18839  | 0.9999      | 0.00108 - 0.00478 |
| Mechanochemical ZIF-9 | 10.93                                 | $\pm 0.0298$                | 76.079 | 0.9999      | 0.05554 – 0.21977 |

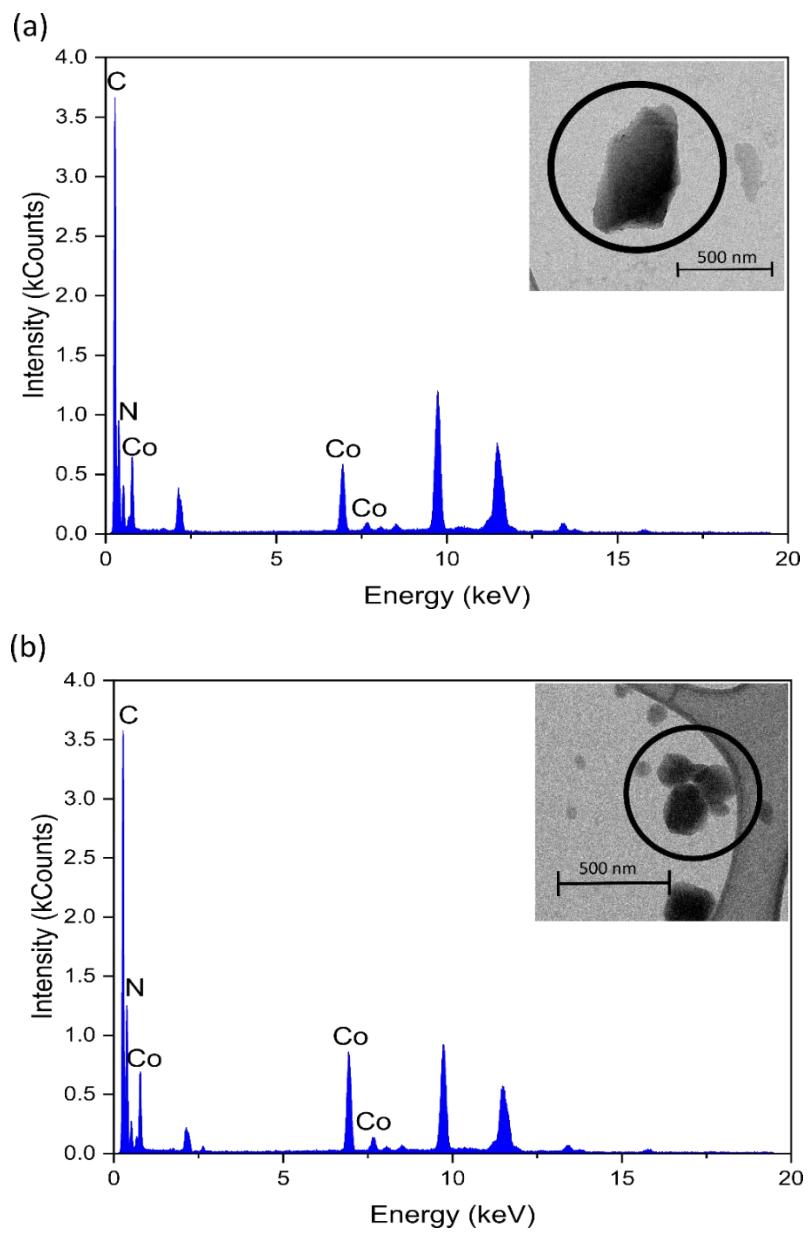


Fig.S.7. EDS spectra for solvothermal synthetized ZIF-9 (a) and mechanochemical synthetized ZIF-9 (b).

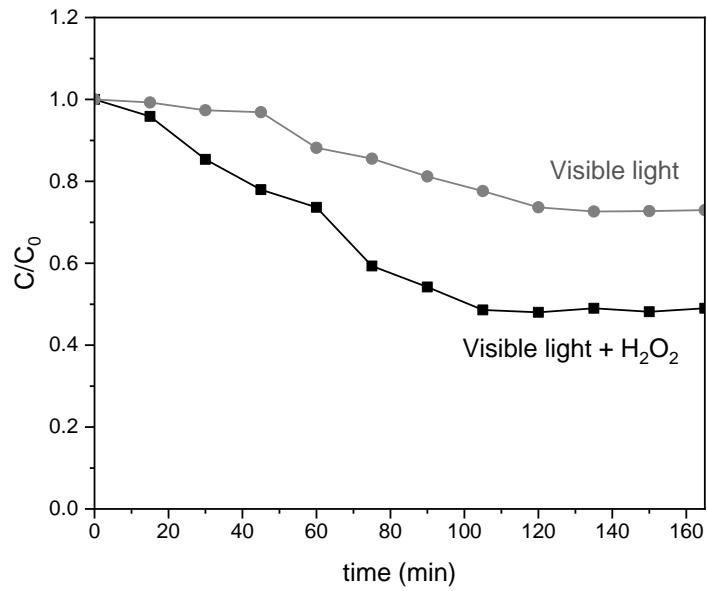


Fig.S.8. Methylene blue degradation by influence of visible light (grey) and the combined action of visible light and  $H_2O_2$  (black).

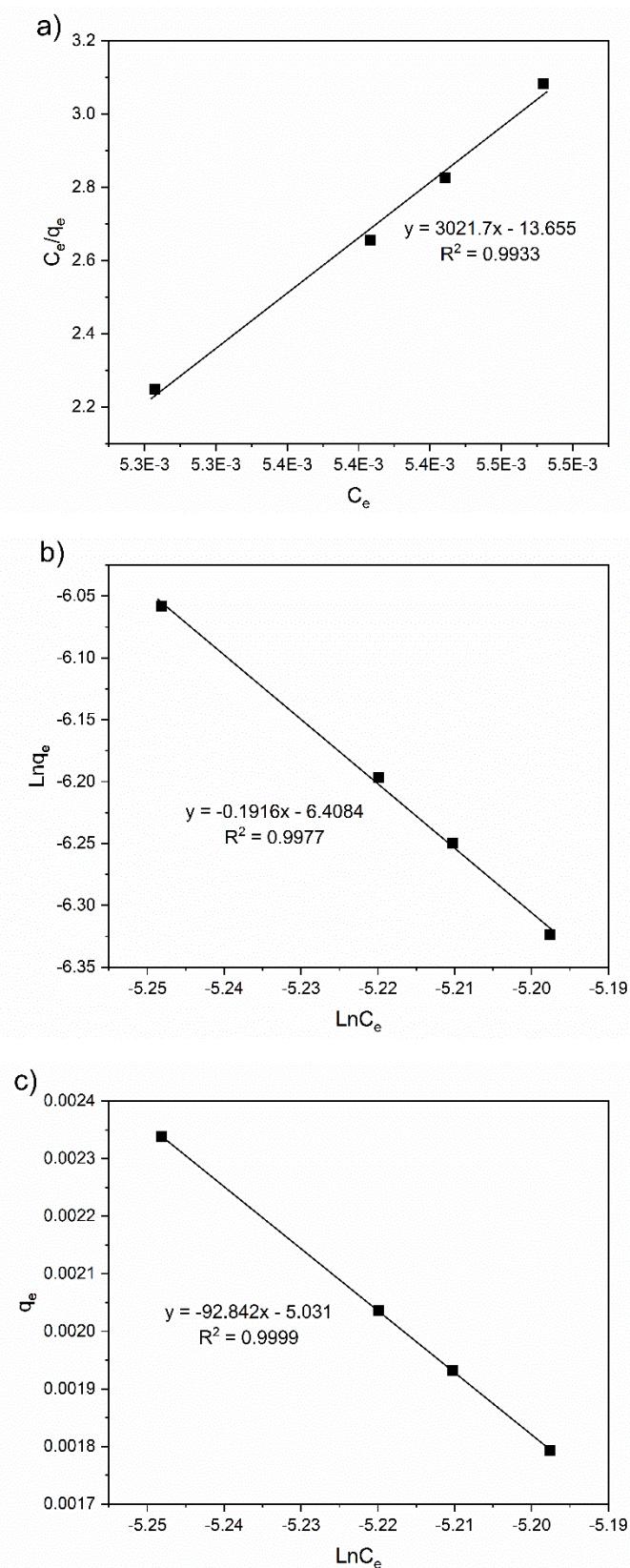


Fig. S.9. MB adsorption on mechanochemical synthesized ZIF-9 based on a) Langmuir, b) Freundlich and c) Tempkin isotherms.

Table S.2. Langmuir, Freundlich and Tempkin model parameters for mechanochemical synthetized ZIF-9.

| Langmuir<br>K <sub>L</sub> (L mg <sup>-1</sup> ) | q <sub>e</sub> (mg g <sup>-1</sup> ) | R <sup>2</sup> | Freundlich<br>K <sub>F</sub> [(mg<br>g <sup>-1</sup> ). (L<br>mg <sup>-1</sup> ) <sup>1/n</sup> ] | n        | R <sup>2</sup> | Tempkin<br>K <sub>T</sub> (L<br>mg <sup>-1</sup> ) | f       | R <sup>2</sup> |
|--|--------------------------------------|----------------|---|----------|----------------|--|---------|----------------|
| -2.213E+02                                       | 3.309E-04                            | 0.9933         | 0.001648  | -5.21921 | 0.9977         | -92.842  | 1.05568 | 0.9999         |

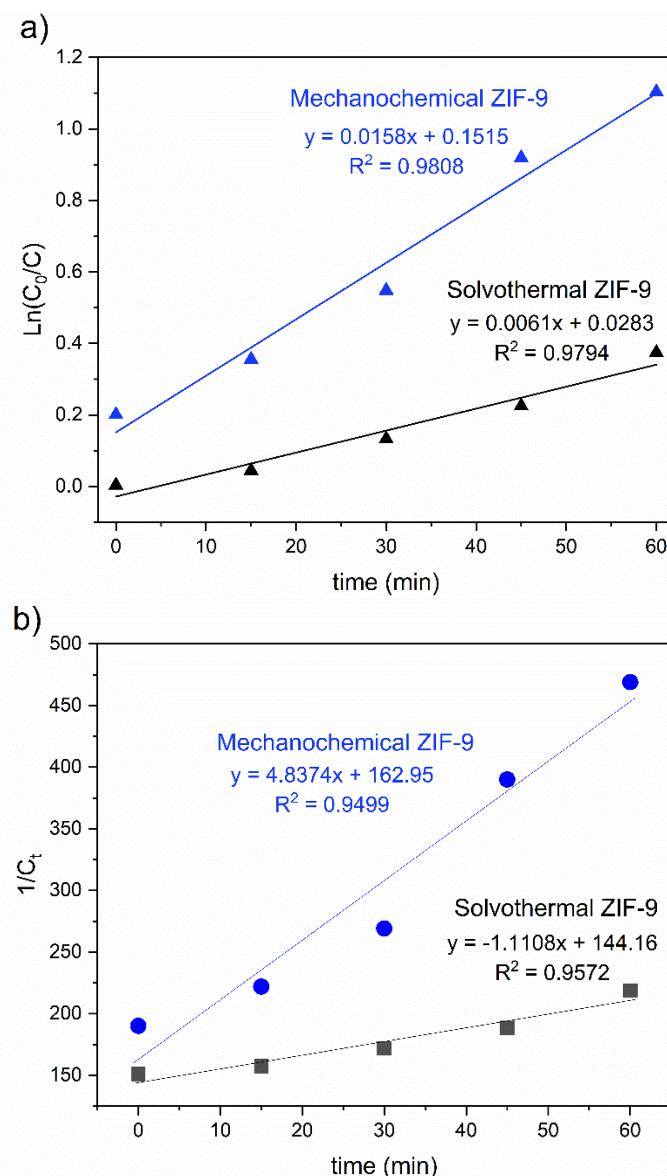


Fig.S.10. Zero-order (a) and pseudo-second order (b) kinetics for solvothermal synthesized ZIF-9 (black) and mechanochemical synthesized ZIF-9 (blue)

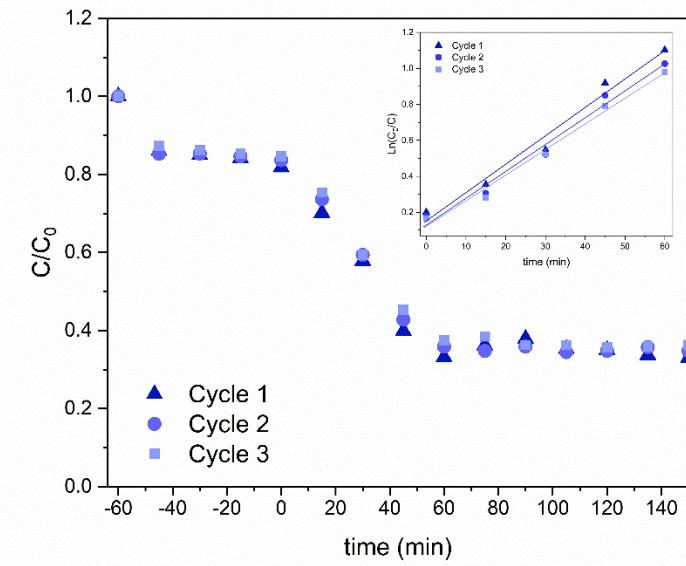


Fig. S.11. Recyclability and first- order kinetic of mechanochemically synthesized ZIF-9 over three cycles.