*Supplementary*

# Sacrificial Zinc Oxide Strategy-Enhanced Mesoporosity in MIL-53-Derived Iron–Carbon Composite for Methylene Blue Adsorption


**Sander Dekyvere [1,2,†], Mohamed Elhousseini Hilal [1,2,3,†], Somboon Chaemchuen [1,2], Serge Zhuiykov [4] and Francis Verpoort [1,2,5,*]**

[1] State Key Laboratory of Advanced Technology for Materials Synthesis and Processing, Wuhan University of Technology, Wuhan 430070, China; sander4a@hotmail.com (S.D.); melhousseinihilal@hkcoche.org (M.E.H.); sama_che@hotmail.com (S.C.)
[2] School of Materials Science and Engineering, Wuhan University of Technology, Wuhan 430070, China
[3] Hong Kong Centre for Cerebro-Cardiovascular Health Engineering (COCHE), Building 17W, Hong Kong Science Park, Hong Kong 999077, China
[4] Department of Solid State Science, Faculty of Science, Ghent University Global Campus, Songdo,
119 Songdomunhwa-Ro, Yeonsu-Gu, Incheon 404-840, Korea; serge.zhuiykov@ghent.ac.kr
[5] National Research Tomsk Polytechnic University, Lenin Avenue 30, 634034 Tomsk, Russia
* Correspondence: francis@whut.edu.cn
† These authors contributed equally to this work.


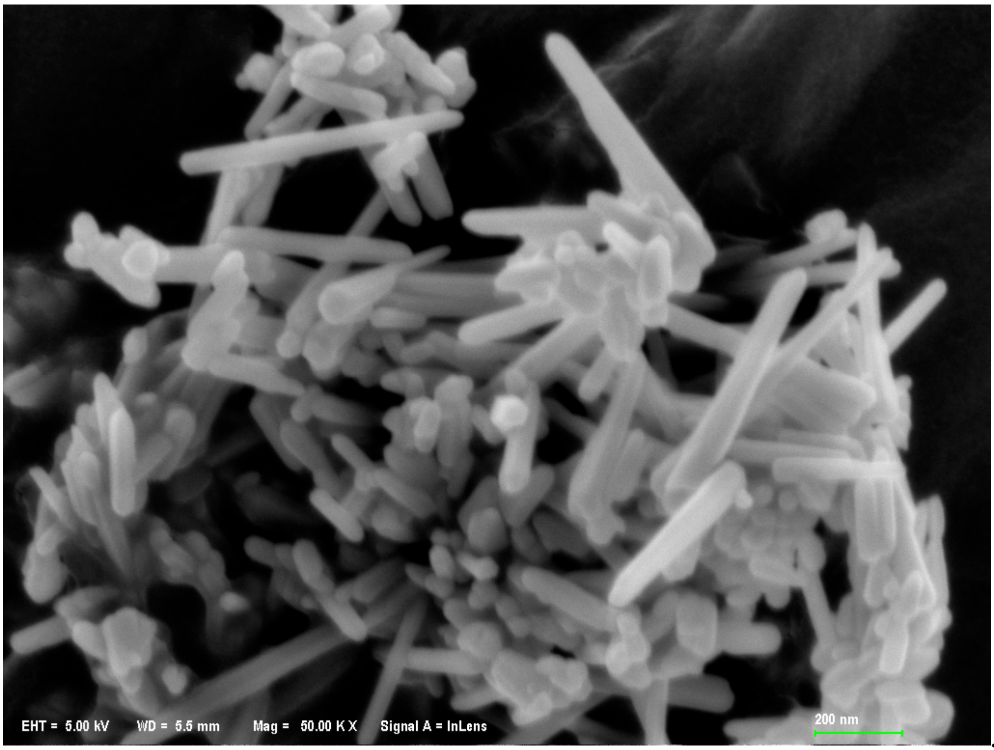


**Figure S1.** SEM micrograph of zinc oxide nanorods.

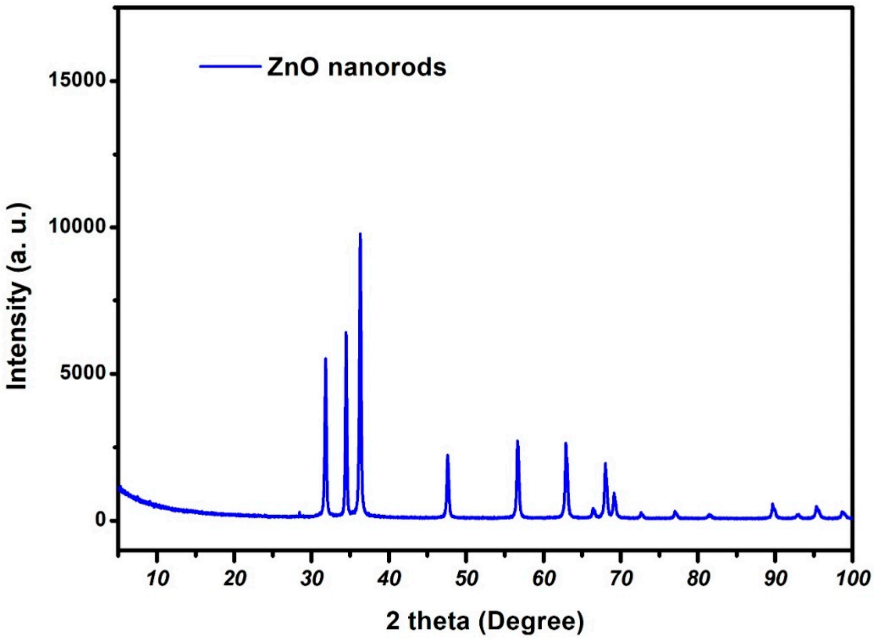


**Figure S2.** X-Ray diffraction of zinc oxide nanorods.

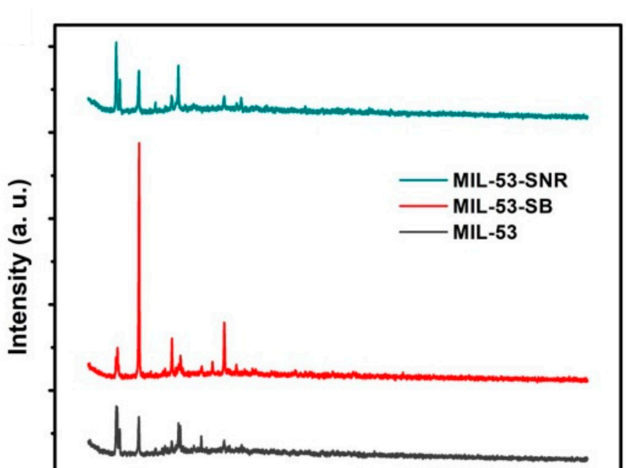


**Figure S3.** X-Ray diffraction spectra of MIL-53-SNR, MIL-53-SB, MIL-53.

**Table S1.** ICP results of MIL-53-SB and MIL-53-SNR.

| | Metallic content before pyrolysis | | Metallic content after pyrolysis | |
|---|---|---|---|---|
| **Sample** | **Fe %** | **Zn %** | **Fe %** | **Zn %** |
| **MIL-53-SB** | 21.77 | 0.26 | 65.75 | 0.01 |
| **MIL-53-SNR** | 21.19 | 0.02 | 61.87 | 0.01 |

**Table S2.** Volume and surface area of micropores and mesopores of pyrolyzed samples.

| | Volume of micropores ($cm^3$ $g^{-1}$) | Surface area of the micropores ($m^3$ $g^{-1}$) | Volume of mesopores (width 2 nm – 20 nm) ($cm^3$ $g^{-1}$) | Surface area of mesopores (width 2 nm – 20 nm) ($m^3$ $g^{-1}$) |
|---|---|---|---|---|
| **Sample** | | | | |
| **Fe-C** | 0.078 | 119 | 0.087 | 47 |
| **Fe-C-SB** | 0.059 | 179 | 0.055 | 27 |
| **Fe-C-SNR** | 0.028 | 78 | 0.169 | 92 |

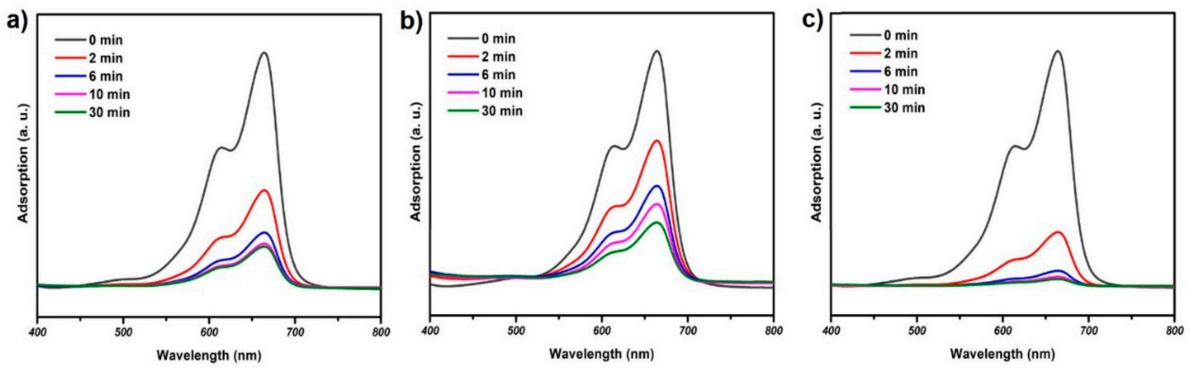


**Figure S4.** Adsorption performance of the three samples: a) Fe-C, b) Fe-C-SB, c) Fe-C-SNR.

**Table S3.** Kinetic parameters of pseudo first order and pseudo second order models for the adsorption of methylene blue onto Fe-C-SNR.

| | Pseudo First Order | Pseudo Second Order |
|---|---|---|
| $q_e$ experimental | 50 | 50 |
| $q_e$ calculated | 35.5 | 50.2 |
| k | $k_1 = 0.5644$ $min^{-1}$ | $k_2 = 10.3322$ g $mg^{-1}min^{-1}$ |
| $R^2$ | 0.9522 | 0.9997 |

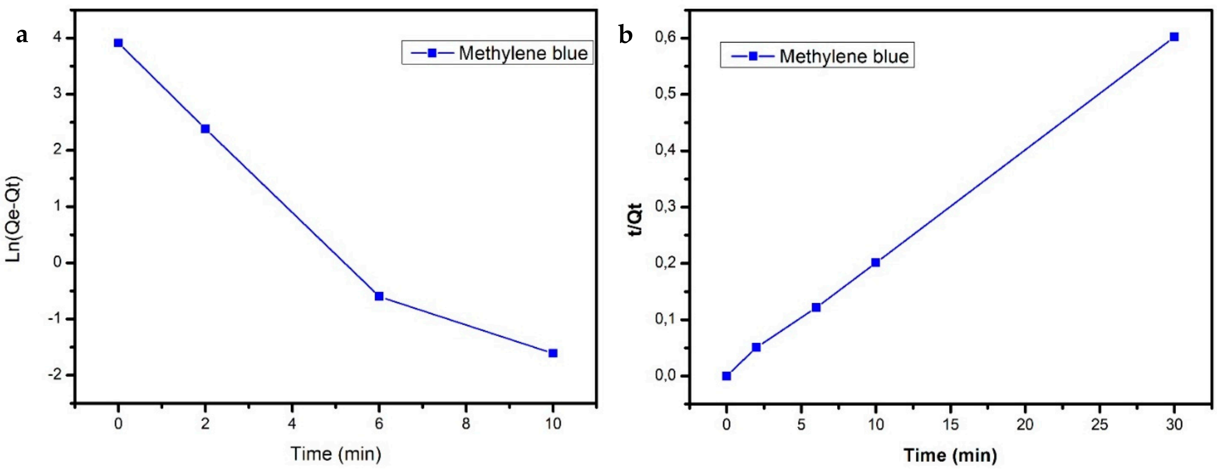


**Figure S5.** Kinetic plots for the adsorption of methylene blue on Fe-C-SNR a) Plot of the pseudo-first-order kinetic model; b) plot of the pseudo-second-order kinetic model.

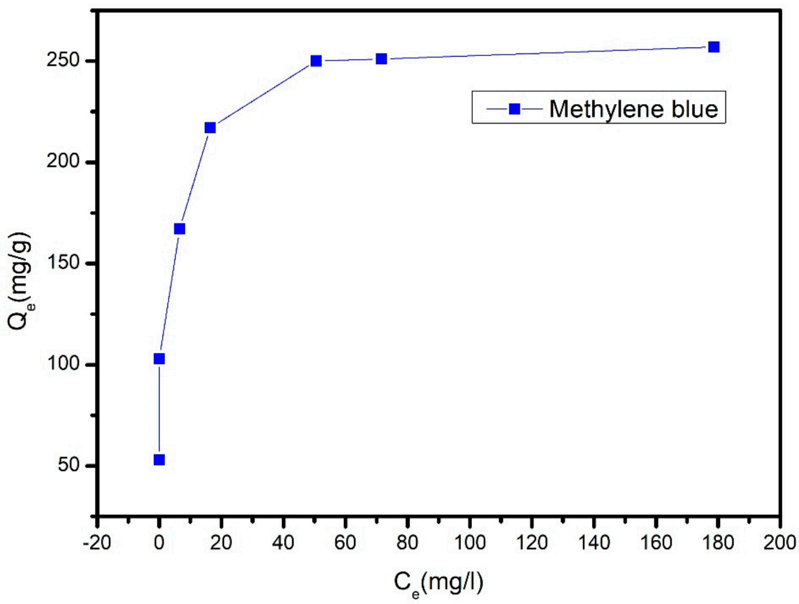


**Figure S6.** Adsorption isotherm of methylene blue on Fe-C-SNR.

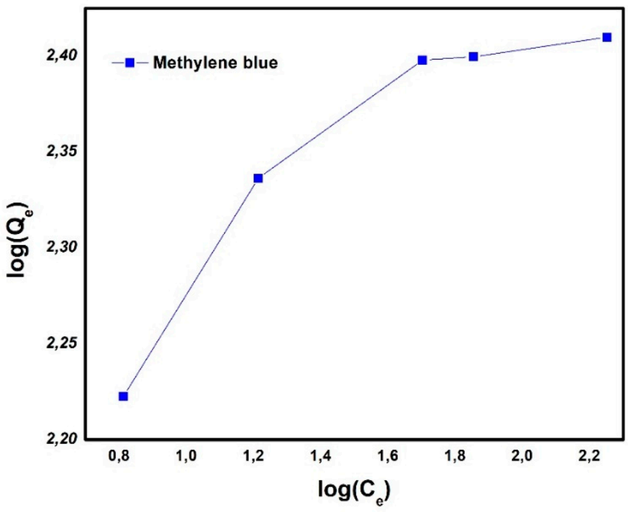


**Figure S7.** Freundlich Linear adsorption isotherm for the adsorption of methylene blue on Fe-C-SNR.

The linear Freundlich adsorption isotherm is given by $\log(q_e) = \log K_F - \frac{1}{n}\log C_e$. Subsequently, the correlation coefficient can be calculated and compared with the Langmuir model.

**Table S4.** Comparitive table of the maximum adsorbed methylene blue from literature.

| | $Q_m$ (mg $g^{-1}$) | Ref |
|---|---|---|
| **Sample** | | |
| **Fe-C-SNR** | 257 | This work |
| **Fe-C** | 125 | This work |
| **Fe-C-SB** | 139 | This work |
| **PF1-1-1-H** | 84 | [1] |
| **Mg-Fe LDH** | 71.94 | [2] |
| **ZVI-GAM** | 151.52 | [3] |
| **nZVI** | 5.53 | [4] |
| **BBCF of 1:2:3:** | 6.8 | [5] |
| **NZVI** | 208.33 | [6] |
| **$Fe_3O_4$@MIL-100(Fe)** | 221 | [7] |
| **$Fe_3O_4$@C NPs** | 117 | [8] |
| **$Fe_3O_4$ carbon composite** | 74 | [9] |

Figure S8 shows the strong peaks of $Fe^0$, with the prominent diffraction peak at 44.6°. It is to note that a slight peak at 35°, matching $Fe_3O_4$ is expectedly emerging due to the adsorption of water in the MIL-53-SNR before the pyrolysis procedure. Effectively, this does not influence the adsorption capabilities of Fe-C-SNR. Moreover, the XRD-spectra display that Fe-C-SNR does not present any noticeable change after the adsorption process.

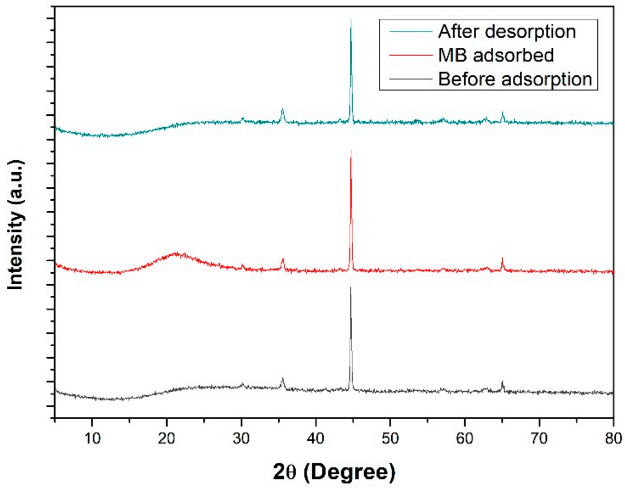


**Figure S8.** X-Ray diffraction spectra of Fe-C-SNR before adsorption, with MB adsorbed, and after desorption.

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