Supporting Information

Characterization and reuse of SiC flakes generated during electrochemical etching of 4H-SiC wafers

Matteo Barcellona⁺^a, Vanessa Spanò⁺^a, Roberto Fiorenza^a, Salvatore Scirè^a, Thomas Defforge^b, Gaël Gautier^b and Maria Elena Fragalà^{*}^a

1 University of Catania and Instm Udr Catania, Department of Chemical Sciences, Viale Andrea Doria, 6, 95125 -

Catania (Italy)

2 GREMAN UMR-CNRS 7347, INSA Centre Val de Loire, Université de Tours, Tours, France

+ Authors equally contributed.

* Corresponding author email : me.fragala@unict.it

1. Methylene blue calibration curve



Fig. S1 Linear fit of methylene blue UV-Vis absorbance as a function of molar concentration. The experimental values are reported in black dots. The linear fit is displayed as a dashed red line.

2. Morphological and surface chemical investigations



Fig. S2 AFM 2D images of 4H-SiC (a) Si-face and (b) C-face flakes. SEM micrographs of 4H-SiC (c) Si-face and (d) C-face flakes sidewalls.

Tab. S1 XPS surface elemental composition of 4H-SiC reference and 4H-SiC flakes investigated on both Si-face and C-face.

Samples	C%	Si%	0%	F%	C/Si	O/Si
4H-SiC Si-face reference	46.99	41.70	11.05	NA	1.13	0.26
4H-SiC Si-face flakes	47.65	36.49	14.14	1.71	1.31	0.39
4H-SiC C-face reference	36.21	37.87	25.92	NA	0.96	0.68
4H-SiC C-face flakes	44.08	39.59	15.01	1.32	1.11	0.38



Fig. S3 Threshold image from SEM micrographs of 4H-SiC (a) Si-face and (b) C-face flakes. The pores are highlighted in white whilst the material portion is displayed in black.

Tab. S2 Average pores' size values of Si-face and C-face 4H-SiC flakes by varying the current density and HF concentration.

	Current density (mA/cm²)	HF concentration (wt%)	Average pores' size (nm)
This work	E10	15	Si-face = 4.4
This work	510	15	C-face = 59.4
Other formulations	E10		Si-face = 23.4
	510	↓ ↓	C-face = 31.3
	1	15	Si-face = 31.8
	\checkmark	15	C-face = 38.7

3. Isotherm adsorption: thermodynamics and kinetics



Fig. S4 Freundlich's thermodynamics model plots from MB adsorption isotherm at 7, 10, 15, 25, and 30 μ M of 4H-SiC (a) Si-face and (b) C-face flakes adsorbents. The linear fit is shown as red dashed line.



Fig. S5 Langmuir's thermodynamics model plots from MB adsorption isotherm at 7, 10, 15, 25, and 30 μ M of 4H-SiC (a) Si-face and (b) C-face flakes adsorbents. The linear fit is shown as red dashed line.



Fig. S6 Pseudo first-order kinetics model from MB adsorption isotherm at 7, 10, 15, 25, and 30 μ M of 4H-SiC (a) Si-face and (b) C-face flakes adsorbents.



Fig. S7 Pseudo second-order kinetics model from MB adsorption isotherm at 7, 10, 15, 25, and 30 µM of 4H-SiC (a) Si-face and (b) C-face flakes adsorbents.



Fig. S8 Weber's kinetics model from MB adsorption isotherm at 7, 10, 15, 25, and 30 μ M of 4H-SiC (a) Si-face and (b) C-face flakes adsorbents.

Adsorbents	MB concentrations (μM)	Pseudo-first order parameters K _{pf} (min ⁻¹), q _e ^f (mg g ⁻¹)	R ²	Pseudo-second order parameters K _{ps} (g mg ⁻¹ min ⁻¹), q _e ^f (mg g ⁻¹)	R ²	Intraparticle diffusion parameters K _{i1} , K _{i2} (mg g ⁻¹ min ^{-1/2}), q _c (mg g ⁻¹)	R ²
	7	K _{pf} =0.079 q _e ^f =4.420	0.999	K _{ps} =0.017 q _e ^f =4.890	0.999	K _{i1} =0.565 K _{i2} =0.0086 q _c =8.066	0.993
4H-SiC flakes Si-face	10	K _{pf} =0.074 q _e ^f =5.386	0.999	K _{ps} =0.013 q _e ^f =5.887	0.999	K _{i1} =0.666 K _{i2} =0.011 q _c =8.139	0.994
	15	$K_{pf} = 0.078$ $q_{e}^{f} = 4.164$	0.999	$K_{ps} = 0.023$ $q_{e}^{f} = 6.980$	0.999	K _{i1} =1.184 K _{i2} =0.026 q _c =5.281	0.981

Tab. S3 Parameters of pseudo-first-order, pseudo-second-order, and Weber's models of MB adsorption kinetics at 7, 10, 15, 25, and 30 μ M on 4H-SiC Si-face and C-face flakes adsorbents.

	25	$K_{pf} = 0.043$ $q_{e}^{f} = 4.084$	0.999	K _{ps} =0.017 q _e ^f =7.666	0.999	K _{i1} =1.183 K _{i2} =0.038 q _c =5.571	0.927
	30	K _{pf} =0.057 q _e ^f =5.877	0.997	K _{ps} =0.011 q _e ^f =9.298	0.999	K _{i1} =1.136 K _{i2} =0.027 q _c =7.097	0.986
4H-SiC flakes	7	$K_{pf} = 0.084$ $q_{e}^{f} = 3.458$	0.945	K _{ps} =0.023 q _e ^f =3.374	0.999	K _{i1} =0.355 K _{i2} =0.016 q _c =8.728	0.993
C-face	10	$K_{pf} = 0.077$ $q_{e}^{f} = 3.333$	0.967	$K_{ps} = 0.023$ $q_{e}^{f} = 3.961$	0.999	K _{i1} =0.464 K _{i2} =0.012 q _c =7.366	0.987

15	$K_{pf} = 0.083$ $q_{e}^{f} = 2.485$	0.870	$K_{ps} = 0.044$ $q_{e}^{f} = 4.490$	0.999	$K_{i1} = 0.208$ $K_{i2} = 0.0004$ $q_c = 16.731$	0.928
25	K _{pf} =0.042 q _e ^f =2.847	0.923	K _{ps} =0.025 q _e ^f =5.758	0.999	K _{i1} =0.967 K _{i2} =0.028 q _c =5.104	0.956
30	$K_{pf} = 0.232$ $q_{e}^{f} = 1.982$	0.981	K _{ps} =0.073 q _e ^f =2.718	0.997	K _{i1} =0.508 K _{i2} =0.002 q _c =4.670	0.994

4. 4H-SiC flakes recovery



Fig. S9 UV-Vis spectroscopy absorbance of SDS recovery treatment of (a) Si-face and (b) C-face 4H-SiC flakes after MB adsorption saturation.



Fig. S10 Bars graph of MB adsorption percentage of Si-face (yellow bars) and C-face (blue bars) flakes before and after recovery performed by HNO₃ 70 wt% cleaning treatment.

5. Molecular sieves application

Tab. S4 List of molecules used in the adsorption investigation of porous 4H-SiC flakes. The label "R" refers to complex cycle systems in the dye molecules. The molecular structures are shown in Fig. S11.

Molecules	Molecule length (nm)	M _w (Da)	Charge	Charged chemical group
мо	1.29	304.34	- (pKa = 3.5) ¹	-R-SO ₃ -
МВ	1.24	284.40	+ (pKa = 8.3) ²	$-R=N^{+}(CH_{3})_{2} \leftrightarrow -R-S^{+}-R-$
RhB	1.29	443.57	+ (pKa = 4.2) ³	-R=N ⁺ (CH ₂ CH ₃) ₂
Rh6G	1.35	443.57	+ (pKa = 6.13) ⁴	-R=N⁺H(CH₂CH₃)
ТВТ	1.10	334.41	+ (fixed)	-N-N*(R)=N-



Fig. S11 Molecular structures of (a) Methylene Blue (MB), (b) Rhodamine B (RhB), (c) Rhodamine 6G (Rh6G), (d) Thiazolyl Blue Tetrazolium (TBT), and (e) Methyl Orange (MO).

References

- 1 R. G. Sandberg, G. H. Henderson, R. D. White and E. M. Eyring, .
- A. Bensedira, N. Haddaoui, R. Doufnoune, O. Meziane and N. S. Labidi, *Polymers and Polymer Composites*, 2022, **30**, 1–16.
- 3 R. Zhang, M. Hummelgrd, G. Lv and H. Olin, *Carbon N Y*, 2011, **49**, 1126–1132.
- 4 S. Rajoriya, S. Bargole and V. K. Saharan, *Ultrason Sonochem*, 2017, **34**, 183–194.