Vortex fluidic mediated synthesis of TiO_2 nanoparticle/MXene composites

Ahmed Hussein Mohammed Al-antaki[a,b], Thaar M. D. Alharbi[a], Suela Kellici[c], Nicholas P. Power[d], Warren Lawrance[e], Colin L. Raston[a*]

a- Flinders Institute for Nanoscale Science and Technology, College of Science and Engineering, Flinders University, Adelaide, SA 5042, Australia.
b- Department of Chemistry, Faculty of Sciences, Kufa University, Kufa, Najaf, Iraq.
c- School of Engineering London South Bank University, 103 Borough Road London, SE1 0AA (UK).
d- School of Life, Health & Chemical Sciences, The Open University, Walton Hall, Milton Keynes, MK7 6AA (UK)
e- College of Science and Engineering, Flinders University, Adelaide, SA 5042.

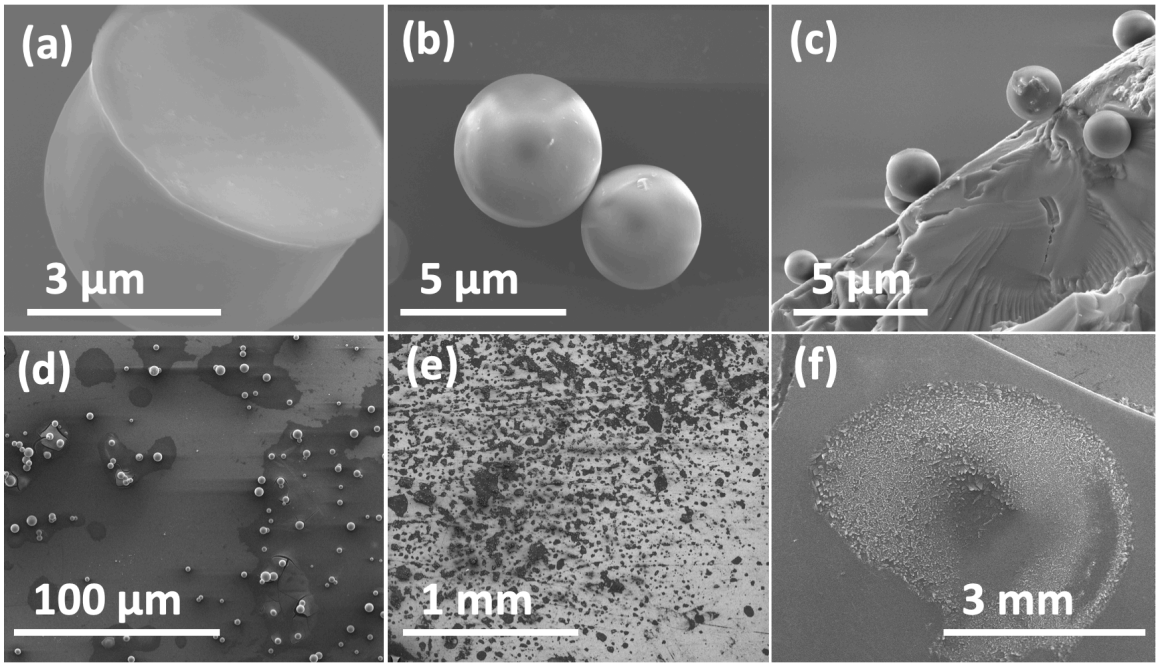

Fig. S1. SEM images of TiO_2NPs/MXene drop cast on silicon wafer and oven dried at 60°C, MXene VFD processing, rotational speed 5k rpm, tilt angle 45° and flow rate 0.75 mL/min. (a-e) TiO_2 NPs/MXene spheres. (f) TiO_2 NPs/MXene sheets.

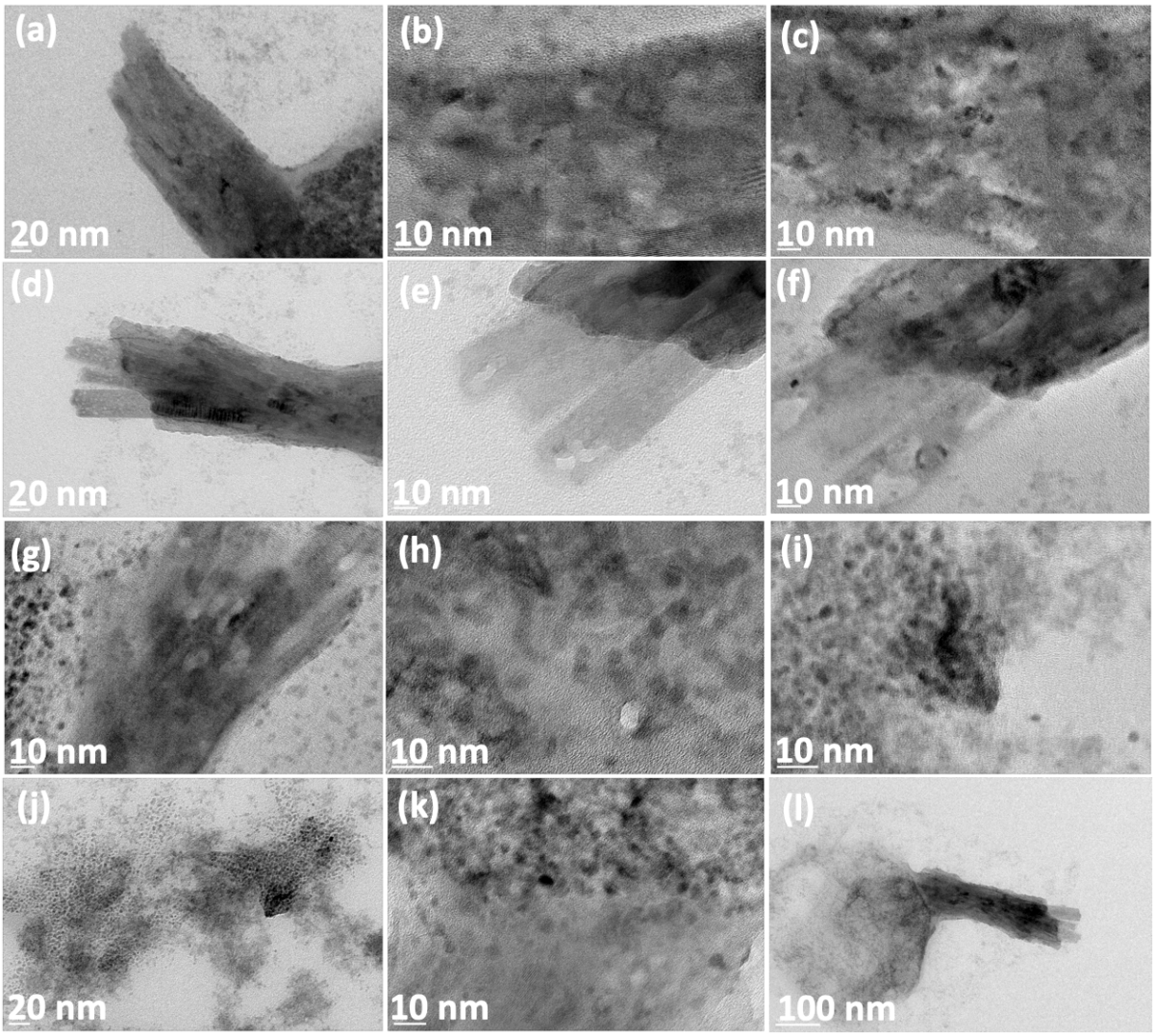

Fig. S2. TEM and HRTEM images for TiO_2 NPs/MXene drop cast on a grid, MXene after 10 mins in a sonic bath (6 kHz) before VFD processing at rotational speed 5k rpm, tilt angle 45^o and flow rate 0.75 mL/min.

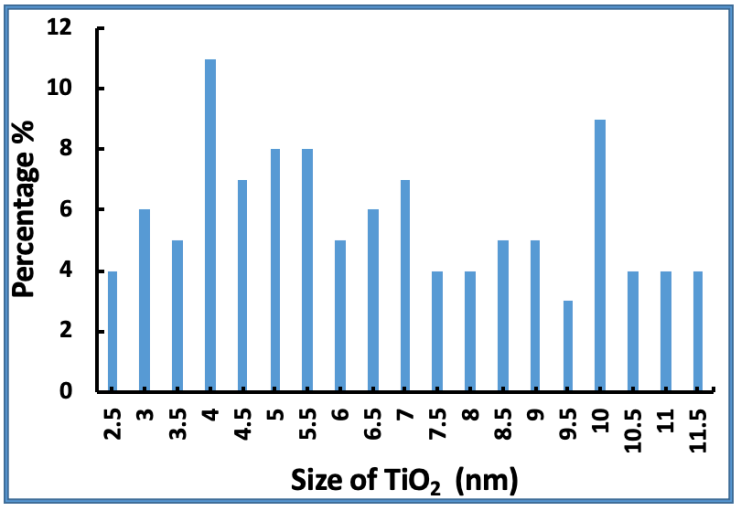

Fig. S3. Account for TiO_2 particle size (109 nanoparticles) in TiO_2 NPs/MXene collected, as derived by HRTEM for MXene VFD processing, rotational speed 5k rpm, tilt angle 45º and flow rate 0.75 mL/min.

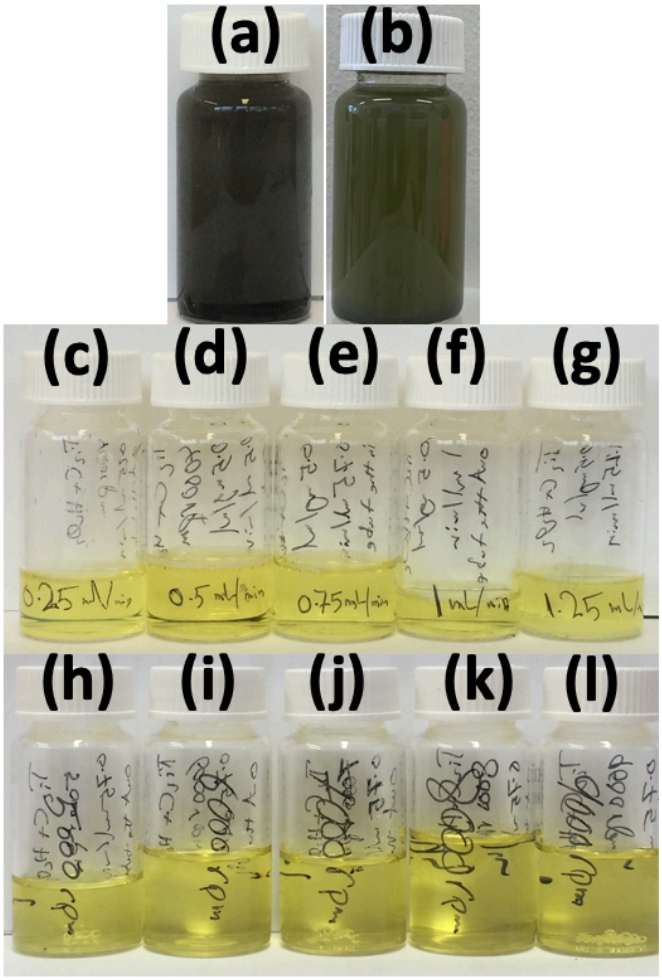

Fig. S4. Photos for (a) MXene in 30% H_2O_2 (0.5 mg/mL). (b) MXene in 30% H_2O_2 (0.5 mg/mL) after 10 mins in a sonic bath (6 kHz). (c-g) TiO_2NPs/MXene product outflow, derived from MXene VFD processing at flow rates 0.25 mL/min, 0.5 mL/min, 0.75 mL/min, 1 mL/min and 1.25 mL/min respectively, rotational speed 4k rpm and tilt angle 45º. (h-l)) TiO_2NPs/MXene product outflow from MXene VFD processing, flow rate 0.75 mL/min, rotational speeds 5, 6, 7, 8 and 9k rpm respectively and tilt angle 45º.

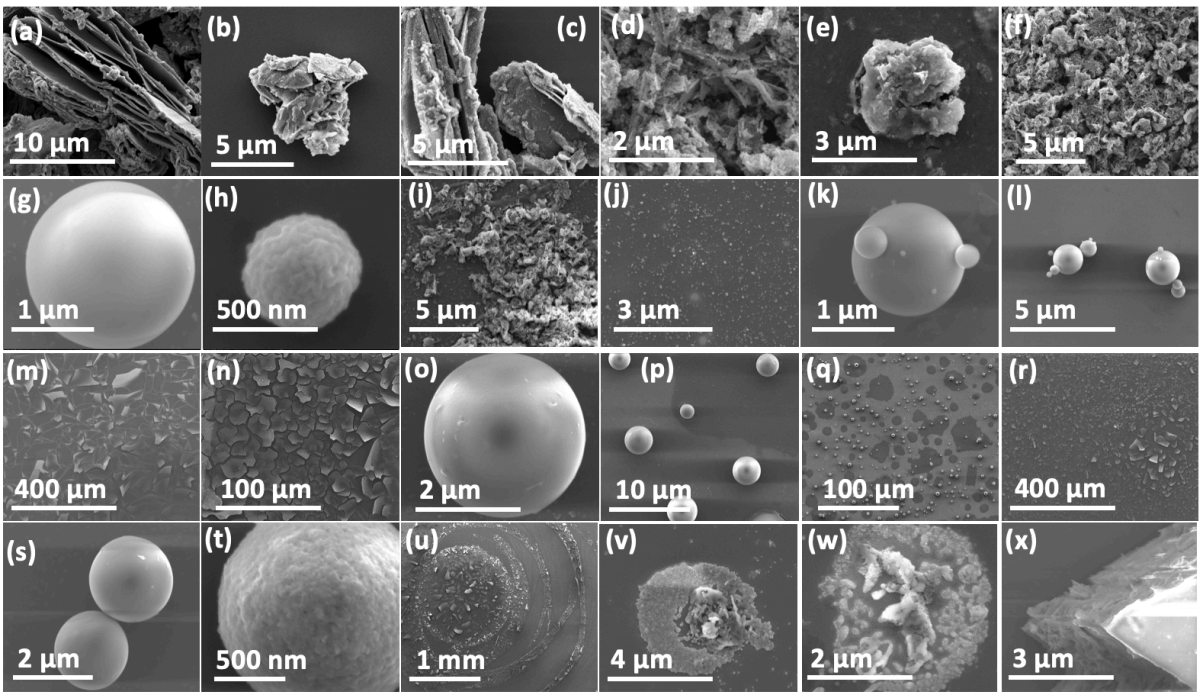

Fig. S5. SEM images of material drop cast on silicon wafers and oven dried at 60ºC. (a-c) MXene as prepared. (d-f) MXene in 30% H_2O_2 (0.5 mg/mL) after 10 mins in a sonic bath (6 kHz). MXene post VFD processing at rotational speed 4k rpm, tilt angle 45º, flow rates (g-j) 0.25 mL/min, (k-n) 0.5 mL/min, (o-r) 0.75 mL/min, (s-u) 1 mL/min, and (v-x) 1.25 mL/min.

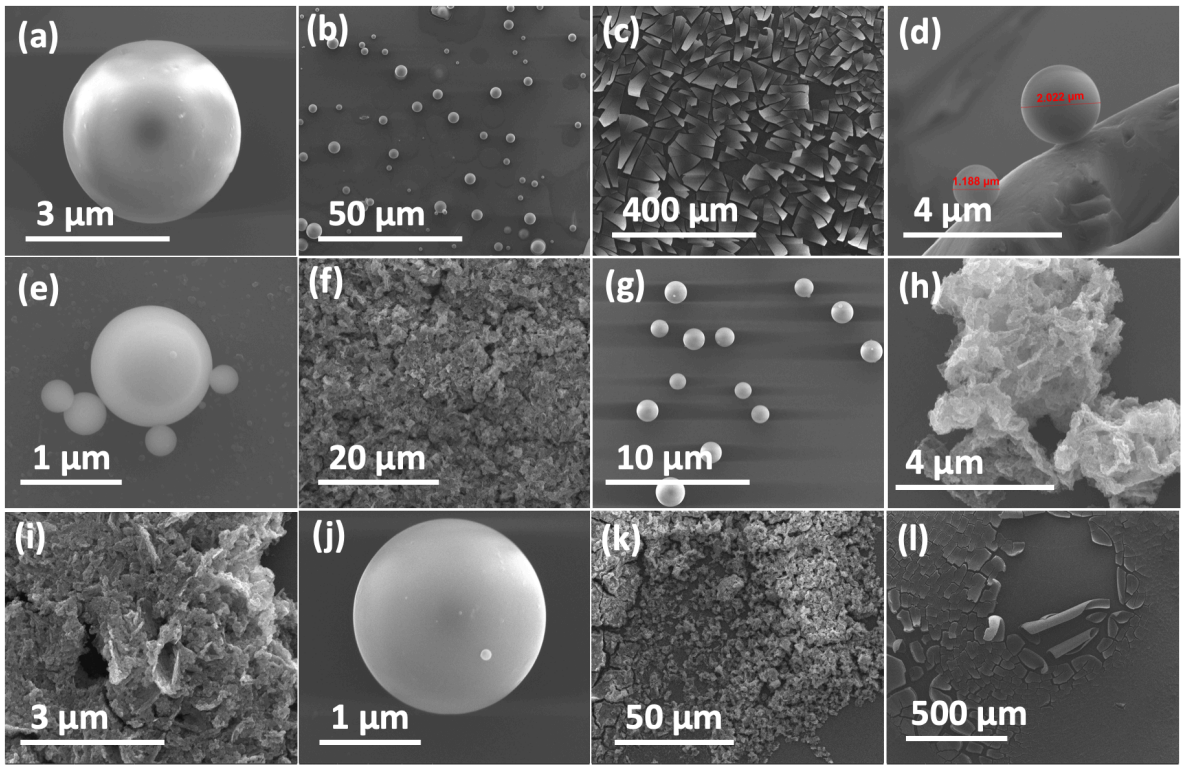

Fig. S6. SEM images for TiO_2NPs/MXene drop cast on silicon wafers and oven dried at 60ºC. VFD processing at tilt angle 45º, flow rate 0.75 mL/min with different rotational speeds, (a-c) 6k rpm, (d-f) 7k rpm, (g-i) 8k rpm, and (j-l) 9k rpm.

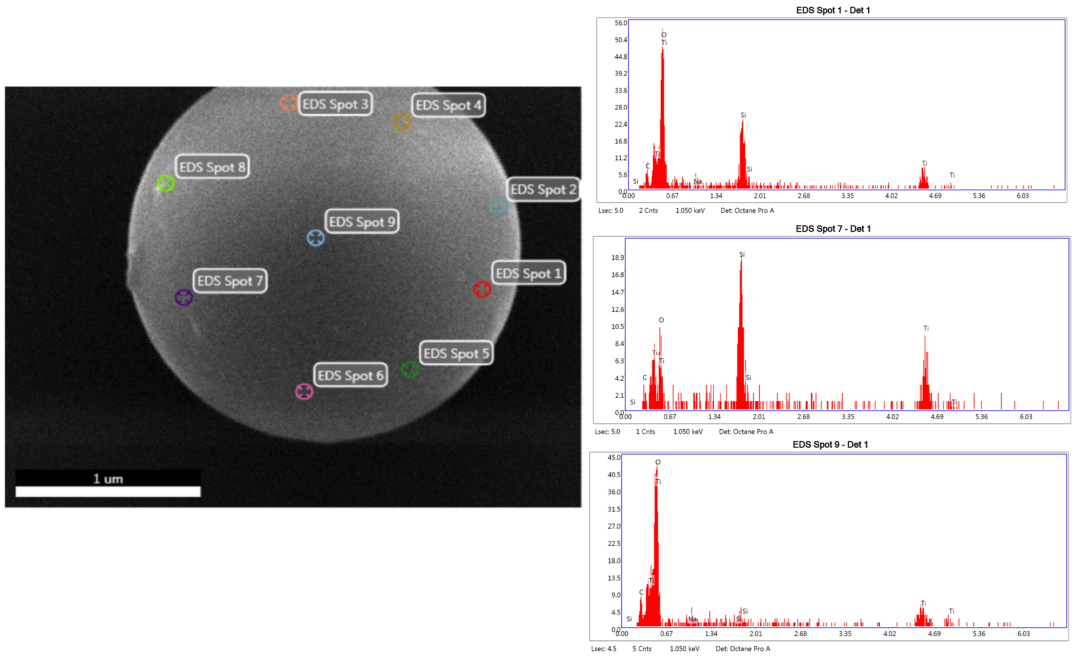

Fig. S7. EDS for TiO_2 NPs/MXene sphere drop cast on a silicon wafer and oven dried at 60°C, optimal VFD processing, tilt angle 45°, flow rate 0.75 mL/min, and rotational speed 5k rpm.

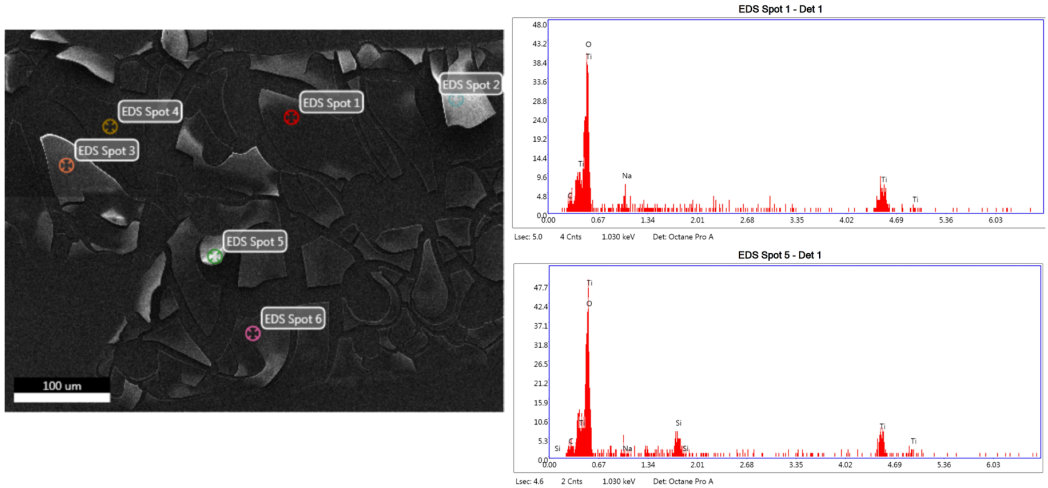

Fig. S8. EDS for TiO_2 NPs/MXene sheets drop cast onto a silicon wafer and oven dried at 60°C, MXene in 30% H_2O_2 (0.5 mg/mL) after 10 mins in a sonic bath (6 kHz) before optimal, tilt angle 45°, flow rate 0.75 mL/min, and rotational speed 5k rpm.

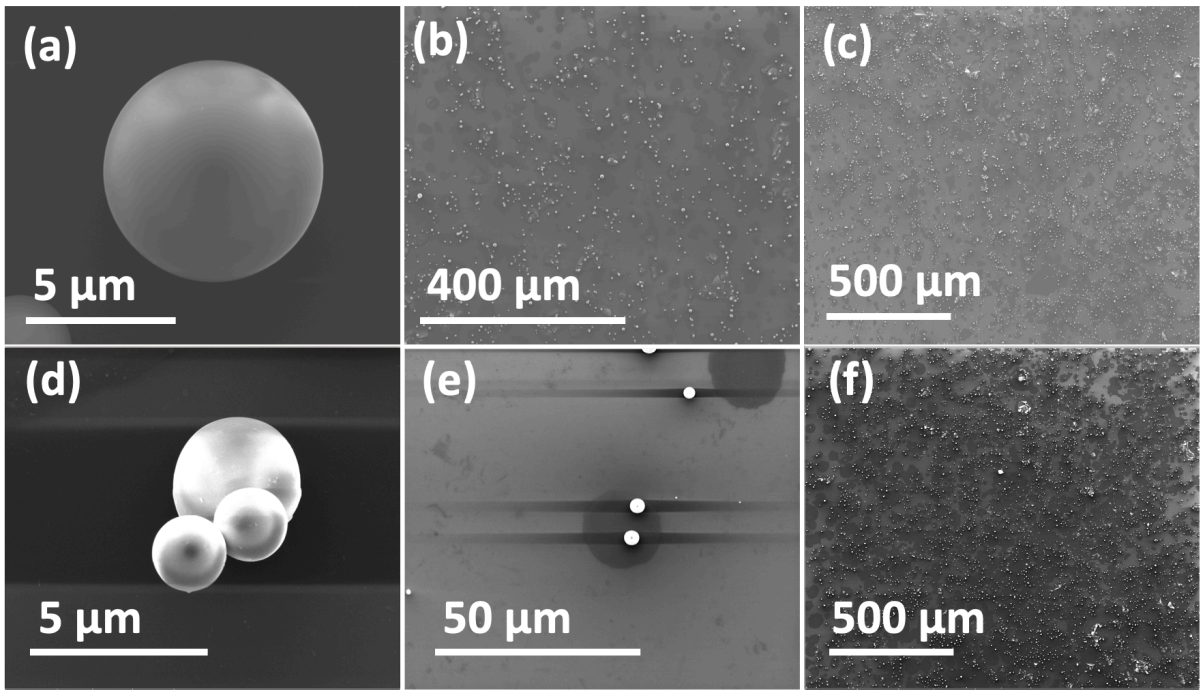

Fig. S9. SEM images for TiO_2 NPs/MXene drop cast onto silicon wafer and oven dried at 60°C, MXene in 30% H_2O_2 (0.5 mg/mL) after 10 mins in a sonic bath (6 kHz) before VFD processing, tilt angle 45°, flow rate 0.75 mL/min, rotational speed 5k rpm. (a-c) Spheres after 25 days for the same sample. (d-f) Spheres after 60 days for the same sample.

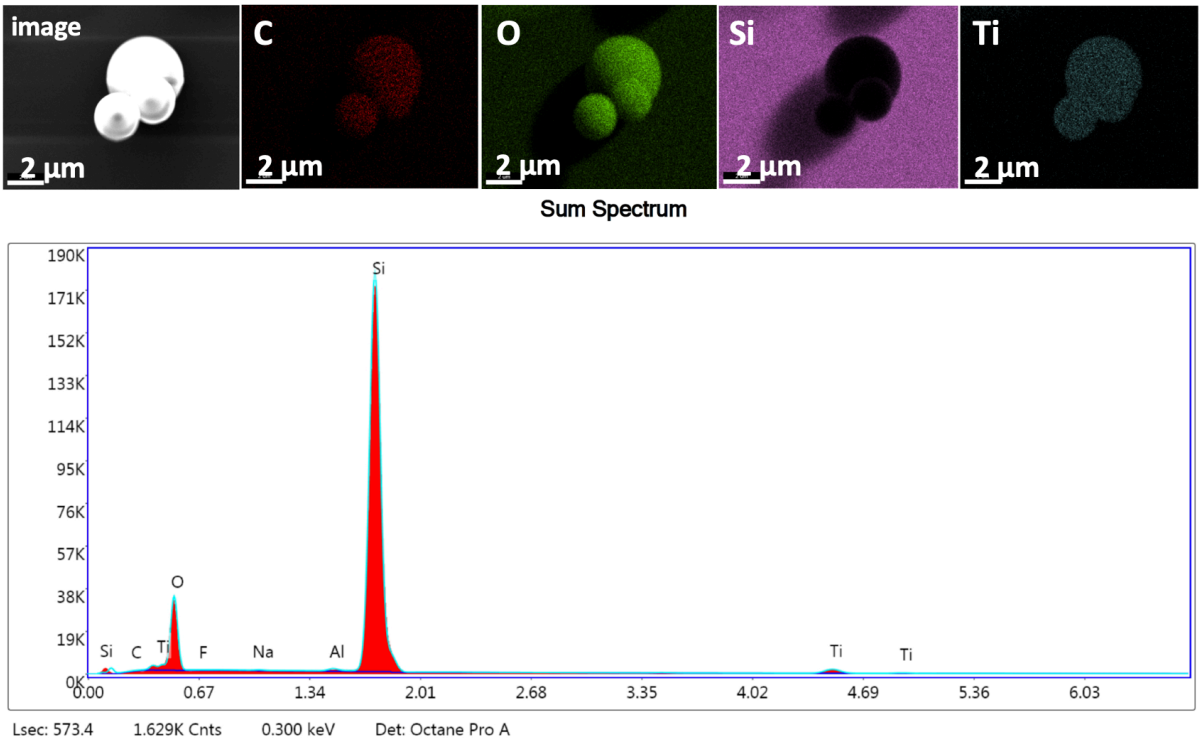

Fig. S10. EDS mapping of stable TiO_2 NPs/MXene spheres drop cast on a silicon wafer and oven dried at 60°C, optimal VFD processing, tilt angle 45°, flow rate 0.75 mL/min, and rotational speed 5k rpm. The spheres are after 60 days of same preparation.

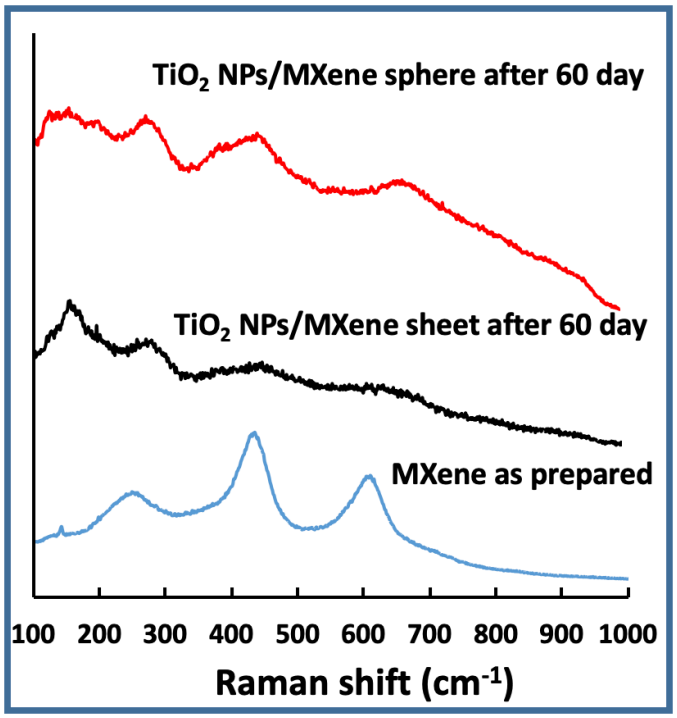

Fig. S11. Raman spectra for TiO_2 NPs/MXene spheres and sheets prepared from MXene optimal VFD processing, rotational speed 5k rpm, tilt angle 45° and flow rate 0.75 mL/min, after 60 days.

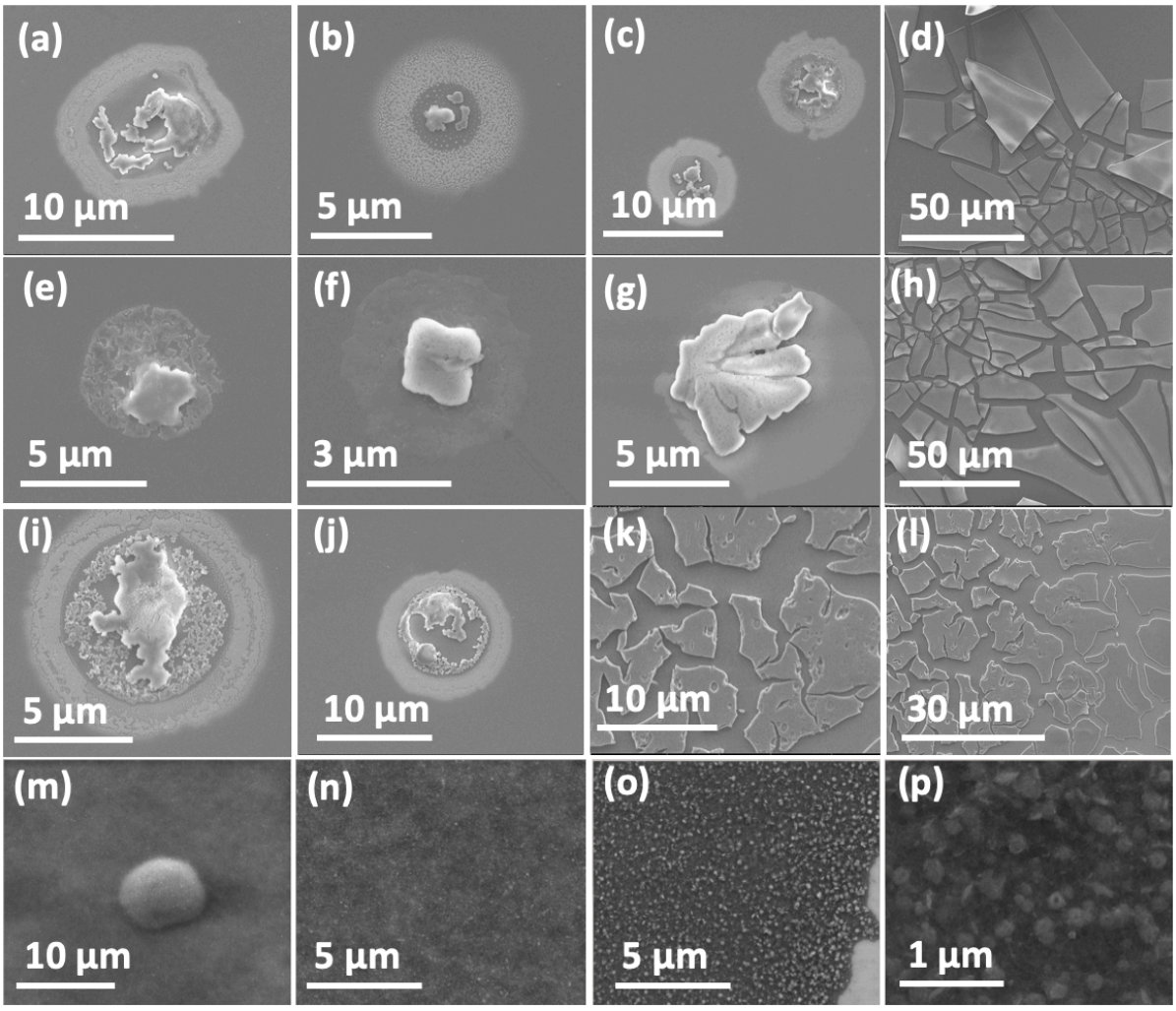

Fig. S12. SEM images in studying the stability of TiO_2NPs/MXene, MXene optimal VFD processing, tilt angle 45°, flow rate 0.75 mL/min, and rotational speed 5k rpm. (a-d) Drop cast material on a silicon wafer after 1 day, (e-h) after 3 days, (i-l) after 6 days, and (m-p) after 60 days.

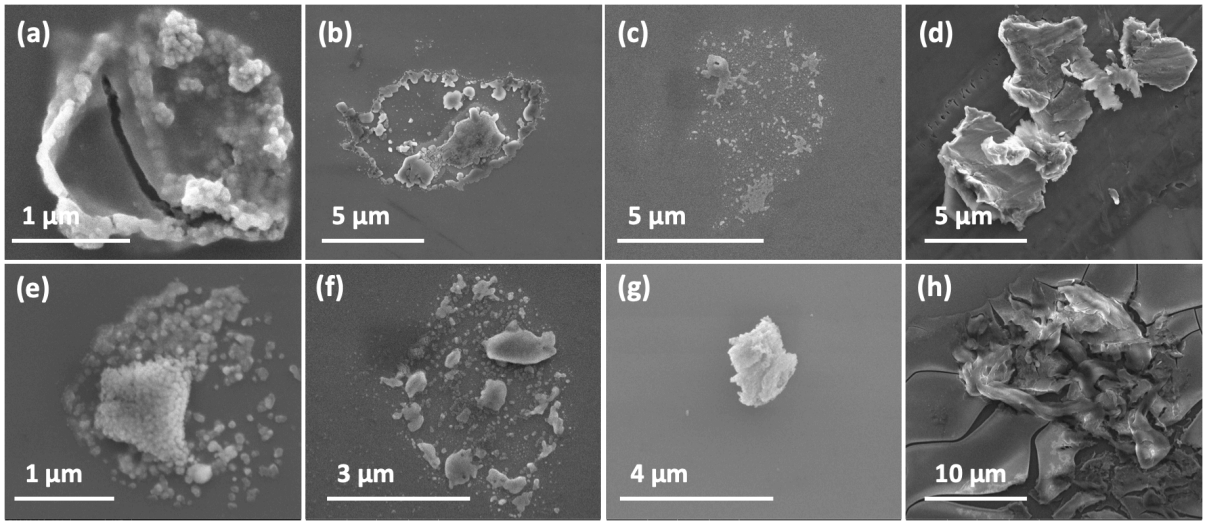

Fig. S13. SEM image for material from control experiments. (a-d) MXene in water (0.5 mg/mL) after 10 min batch sonication (6 kHz) before delivering to the VFD at a flow rate of 0.39 mL.min^{-1} with 30% H_2O_2 added at the same time through a second jet feed at a flow rate of 0.39 mL/min, rotational speed 5k rpm, and tilt angle 45°. (e-h) MXene in water and 30% H_2O_2 volume ratio 5:1, concentration of MXene 0.5 mg/mL with the solution previously bath sonicated for 10 min (6 kHz), flow rate of solution was 0.75 mL/min, rotation speed 5k rpm, and tilt angle 45°.

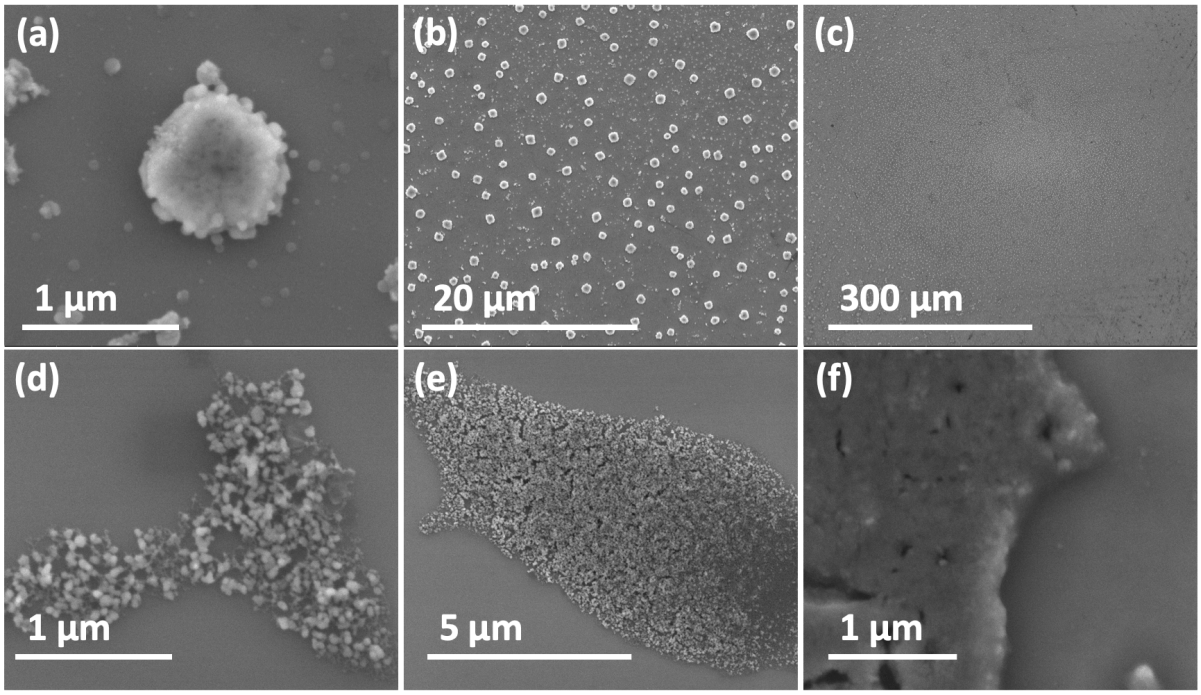

Fig. S14. SEM images for TiO_2 NPs < 100 nm in diameter in 30% H_2O_2 (0.5 mg/mL) after optimal VFD processing, tilt angle 45°, flow rate 0.75 mL/min, and rotational speed 5k rpm.

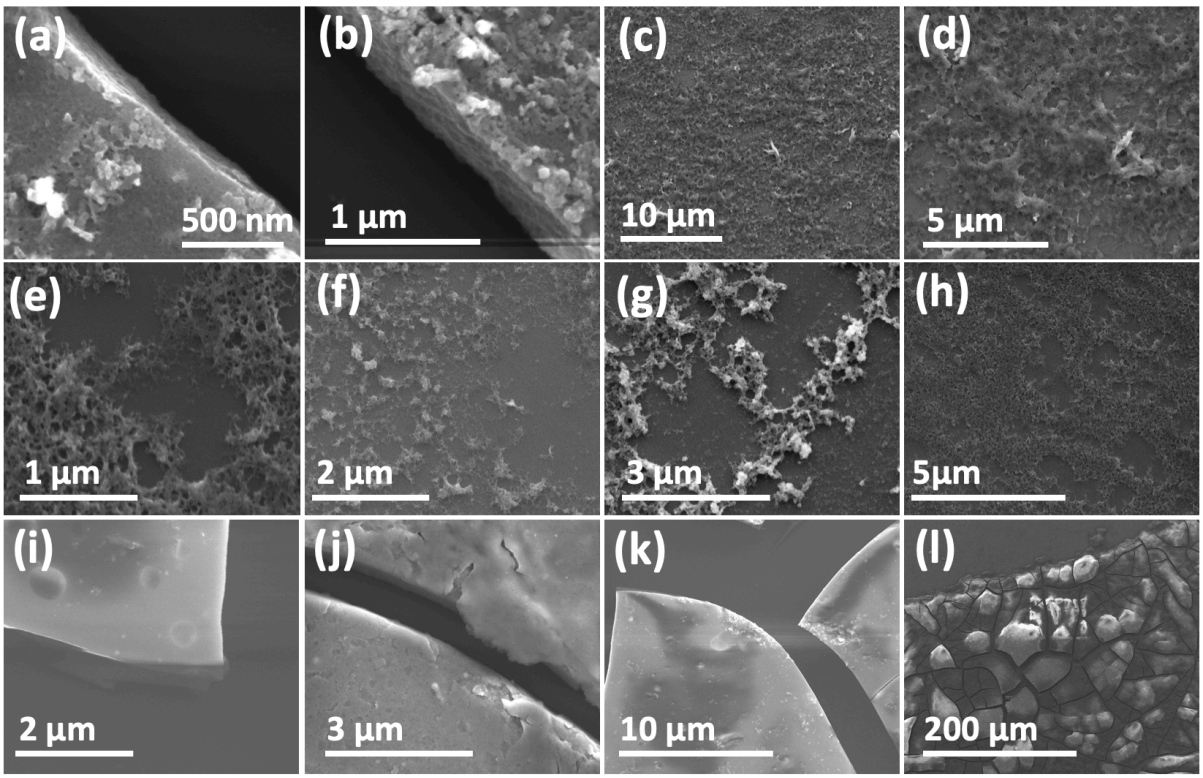

Fig. S15. SEM images for (a-d) MXene in 30% H_2O_2 + DMF ratio 1-1, then 10 mins of bath sonication (6 kHz) before VFD processing, rotational speed 5k rpm, concentration 0.5 mg/mL, the flow rate 0.75 mL-min and tilt angle 45º. (e-h) Collected TiO_2NPs/MXene from the tube after rotating at 5k rpm, 0.75 mL/min, 0.5 mg/mL with a tilt angle of 45º, re-dispersed in 15 mL of DMF previously passed through the VFD, rotational speed 8k rpm, flow rate 0.75mL/min, tilt angle 45º. (i-l) MXene in 30% H_2O_2 (0.5 mg/mL) after 10 mins bath sonication (6 kHz) before passing through the VFD with another jet feed delivering DMF, tilt angle 45º, rotational speed 5k rpm and flow rate for both MXene in 30% H_2O_2 and DMF 0.38 mL/min.