

Electronic Supplementary Information (ESI)

**Mesoporous titanosilicate/reduced graphene oxide composites:
layered structure, high surface-to-volume ratio, doping effect
and potential application in dye-contaminated removal**

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1. Preparation of graphene oxide (GO)

Graphene oxide was synthesized from expanded graphite by a modified Hummers method [W.S. Hummers and R.E. Offeman, *J. Am. Chem. Soc.*, 1958, **80**, 1339]. A desirable amount of expandable graphite (grade 1721, Asbury Carbon) was heated for 10 s in a microwave oven, gaining 150 times the volume of its original one. The three-necked flask containing 500 mL of concentrated H₂SO₄ was chilled in the ice bath to 273 K and 5 g of expanded graphite were gradually added under mechanically stirring. 30 g of KMnO₄ was slowly added so that the temperature did not exceed 293 K. The temperature was then elevated to 308 K and the suspension was stirred for 2 h.

The flask was subsequently chilled again in the ice bath and 1 L of deionized water was slowly added to maintain the temperature below 343 K. The mixture was stirred for 1 h, diluted and added with 50 mL of H₂O₂ (30 wt%). Vigorous bubbles appeared as the color of suspension changed from dark brown to yellow. The suspension was centrifuged and washed with aqueous HCl solution (10%), followed by centrifuging at 10,000 rpm and washing with deionized water to entirely removed the acid until the pH of GO dispersion reached 6. The as-synthesized GO dispersion was in the paste form and the concentration of GO was 12.5 mg mL⁻¹, which was determined by vacuum-drying at 353 K for 24 h.

2. Supplementary figures

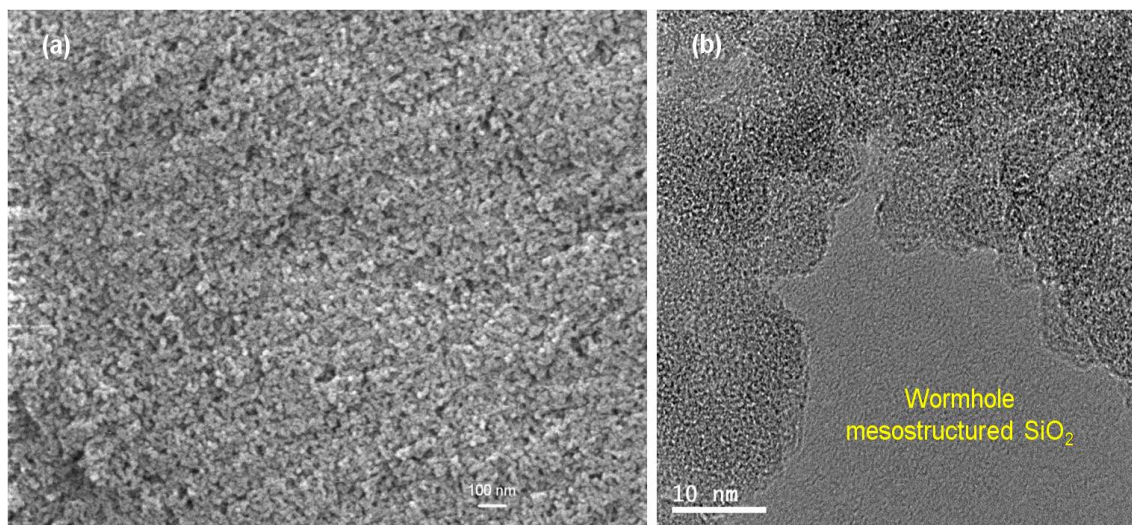


Fig. S1. FE-SEM and HR-TEM images of pure MS.

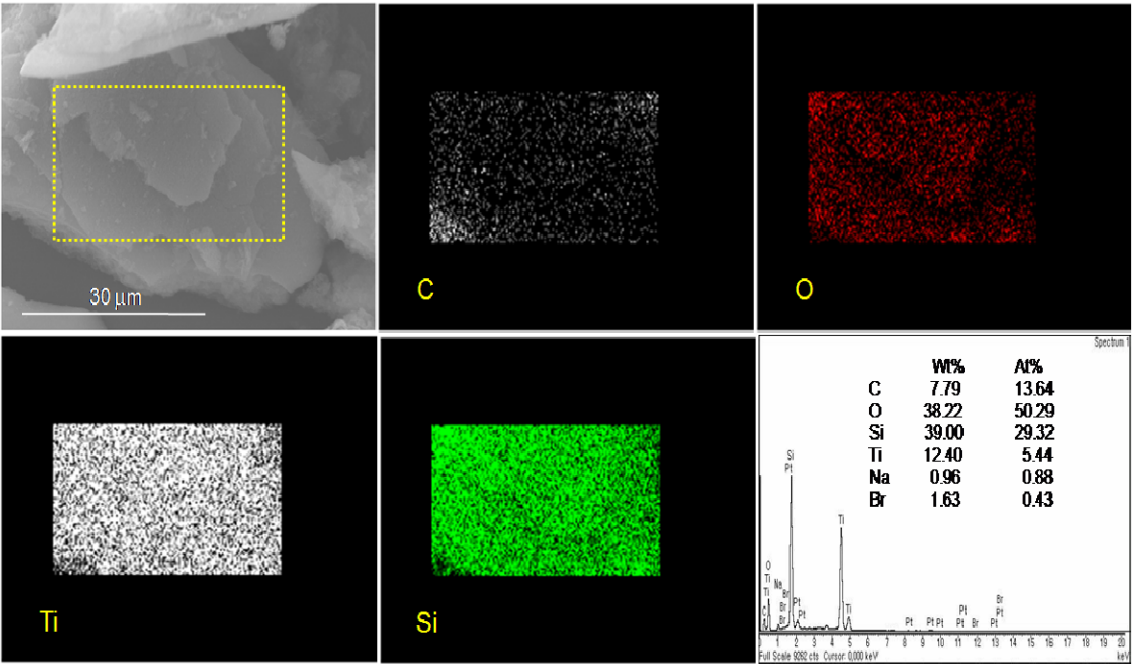


Fig. S2. Composition analysis of MTSG-20 composite by FE-SEM/EDX and mapping.

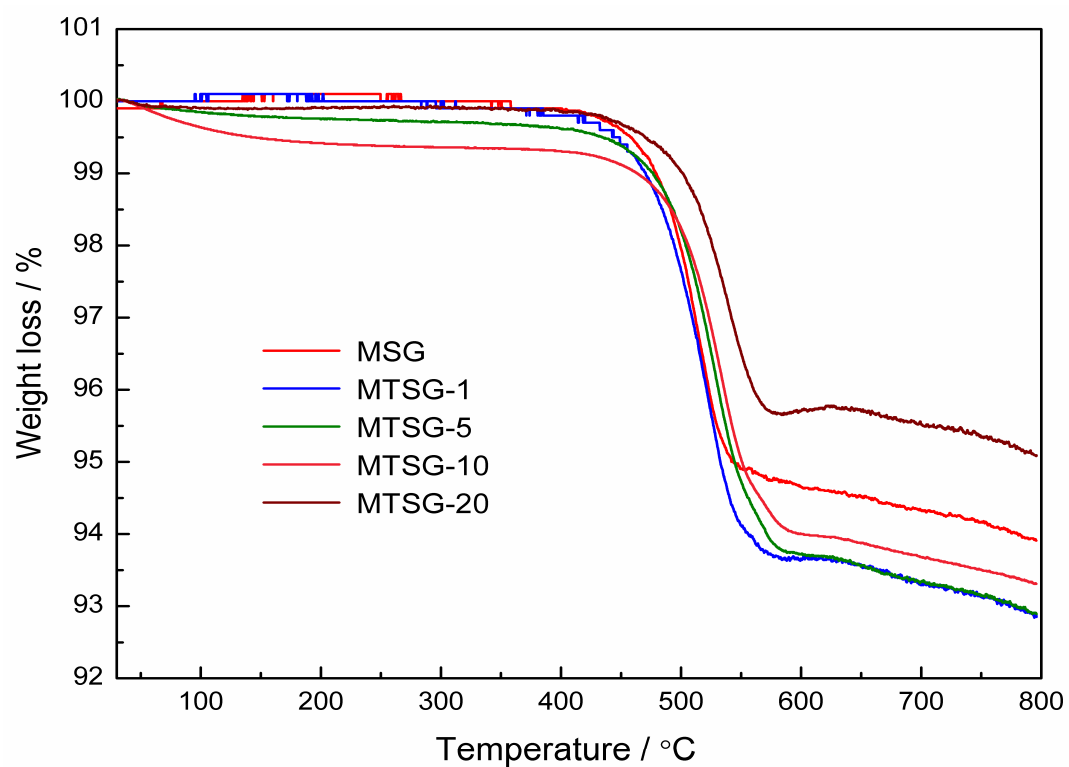


Fig. S3. TGA analysis of MSG and MTSG-*i* composites (in the flow of N₂/air, 60/40 ml min⁻¹).

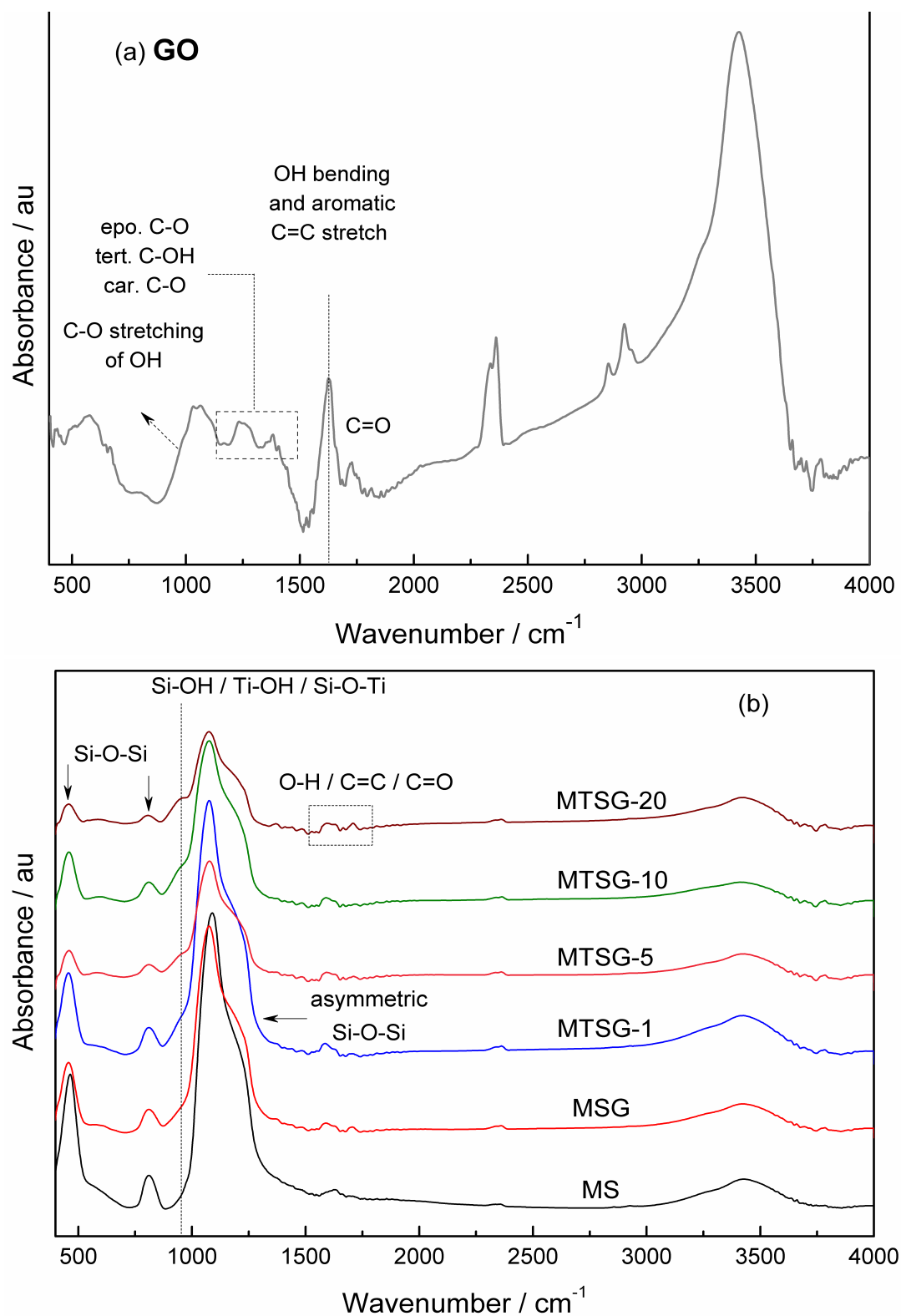


Fig. S4. FT-IR spectra of (a) GO and (b) MS, MSG and MTSG-*i* composites.

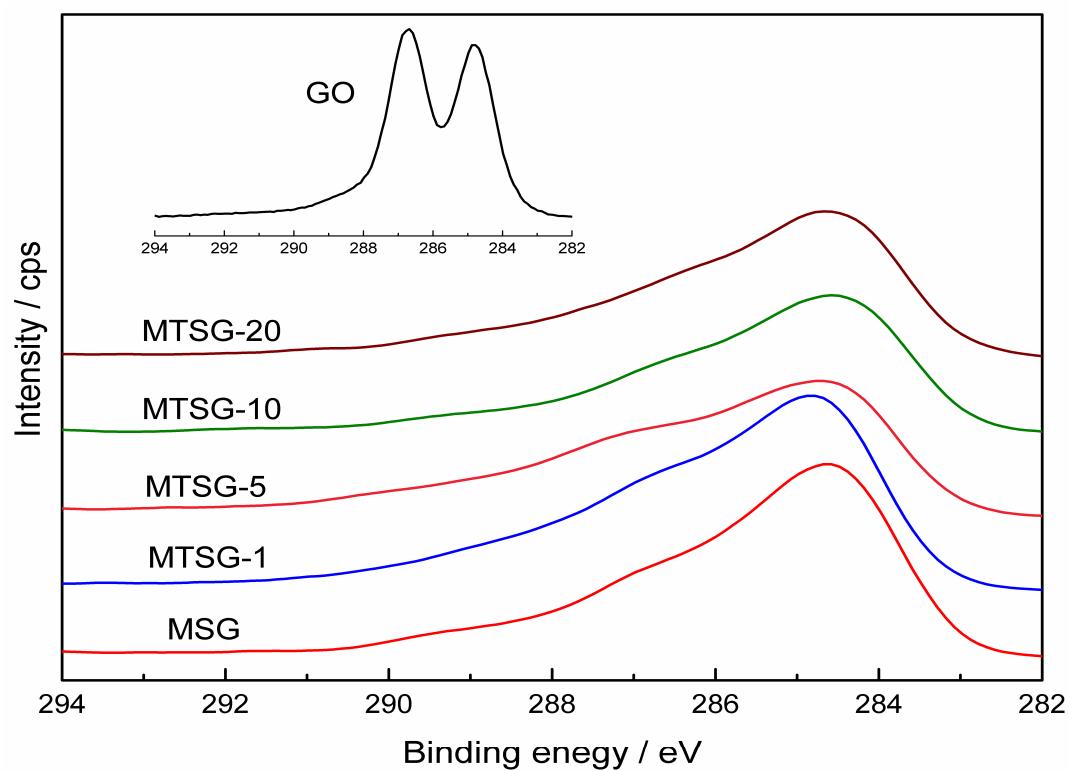


Fig. S5. XPS C 1s core-level spectra of GO, MSG and MTSG-*i* composites analysis.

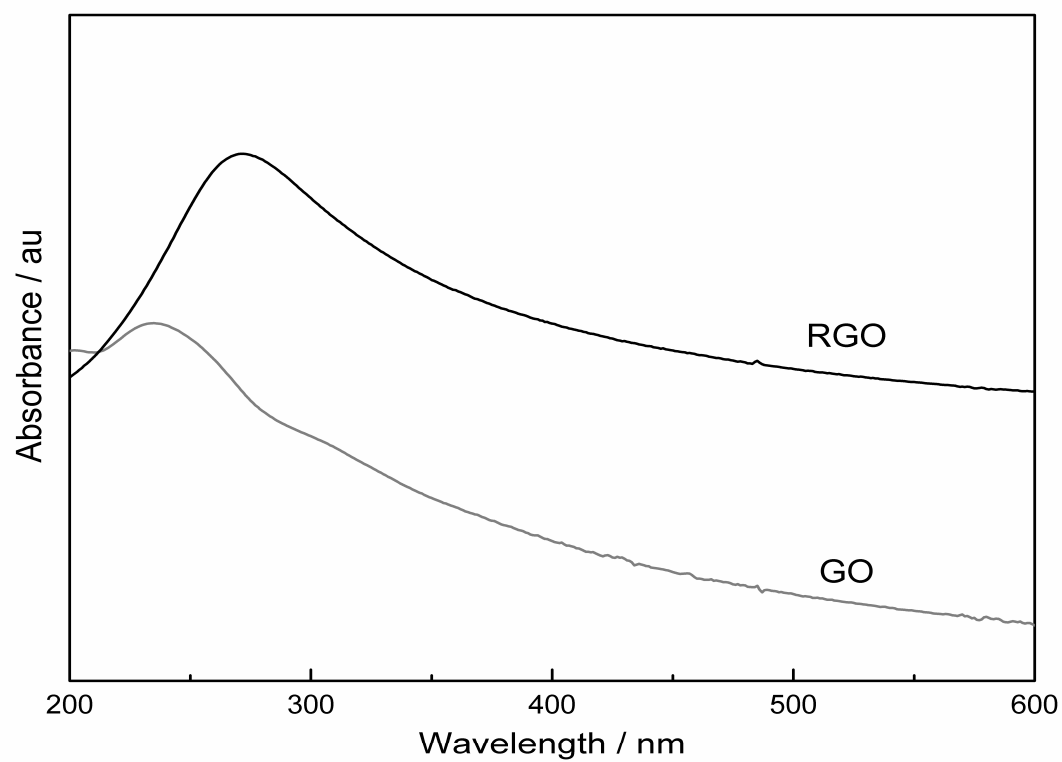


Fig. S6. UV-Vis-DRS spectra of GO and RGO.

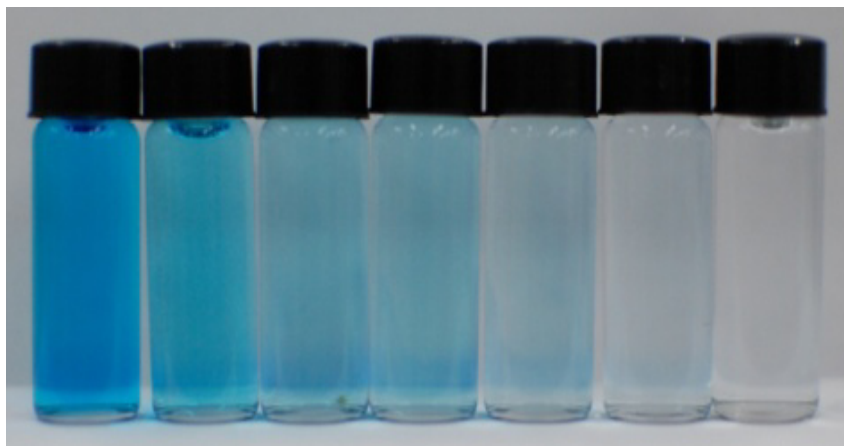


Fig. S7. Optical photograph for aqueous methylene blue solution before and after degradation by the IPCAs.

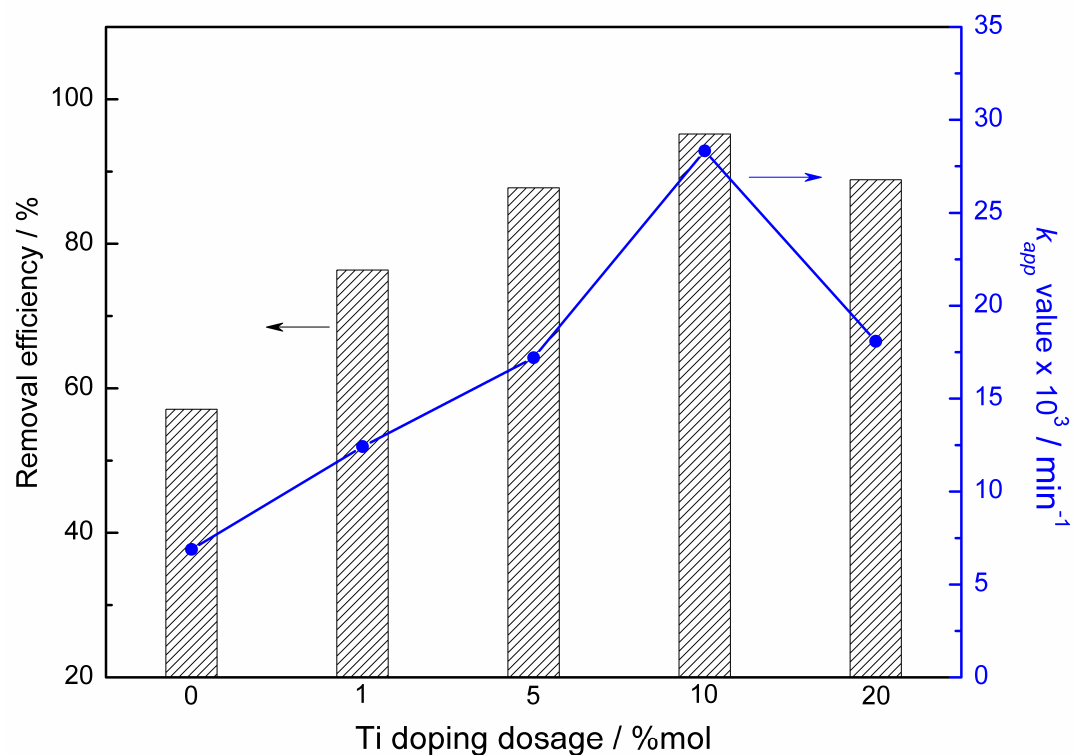


Fig. S8. Comparison of dye removal efficiencies and apparent rate constants of all composites with different Ti dosages.

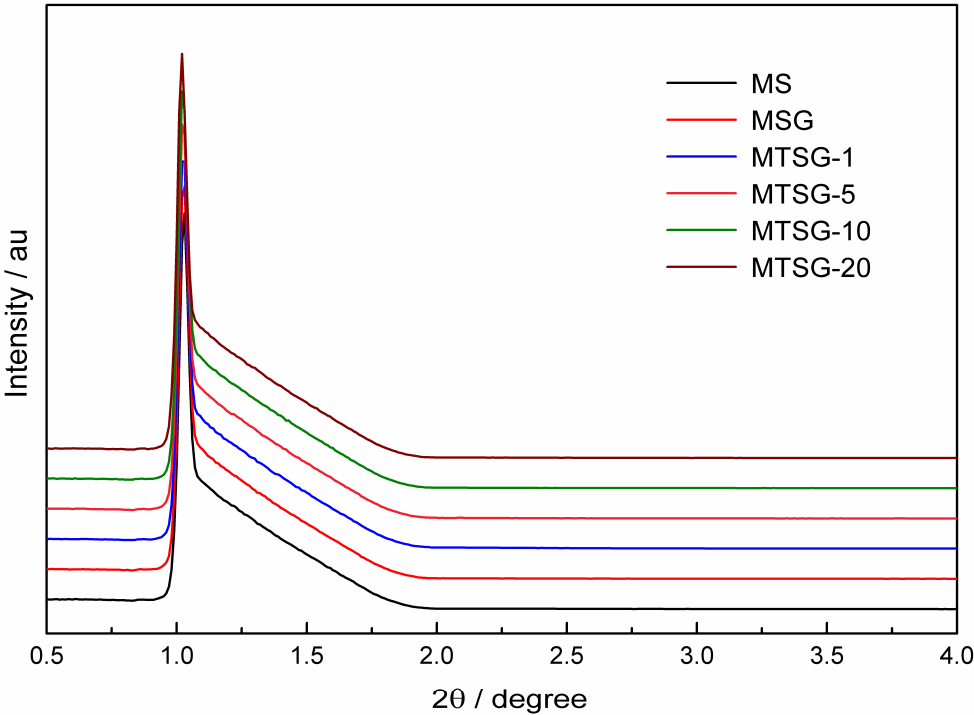


Fig. S9. Small angle XRD patterns of MS, MSG and MTSG-*i* composites.