Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supplementary Information

Green synthesis of mesoporous flower-like TiO_2/Graphite nanosheets (TGNS) prepared by high-pressure homogenization (HPH)

Dynamic light scattering (DLS) study

From the dynamic light scattering images exhibit the TiO_2 particle size distribution information of all the TGNS samples, there are two main particle sizes at around 100-1000 nm for the ultrasonic supernatant of the TGNS samples from A to D, but there exists wide particle size distribution for TGNS-E. This phenomenon could be explained by considering that there are different stages of depositing TiO_2 on the surface of GNS, which can produce different particles possessing diverse sizes. For TGNS composites from A to D, the particle size distribution is very uniform at 100-1000 nm. As TiO_2 ratio increases, there will be a small residual percentage of Ti^{3+} aggregate themselves instead of forming crystals on the surface of GNS and that is the reason of two main particle sizes for TGNS samples from A to D , although the most component (80 %-90 %) of TGNS is still GNS loaded with flower-like TiO_2 particles uniformly distributed. When TiO_2 ratio is too big for them depositing on the GNS surface, there will be huge amount of remanent TiO_2 particles aggregate themselves, TGNS-E tends to form particles with different sizes which are decided by the level that $TiCl_3$ hydrolyzes in liquid solution. Besides, from the DLS pictures, as TiO_2 ratio in the composites increases, there is a clear particle size shift from 110 nm to 1000 nm, which indicates that TiO_2 has been gradually formed on the surface of GNS.

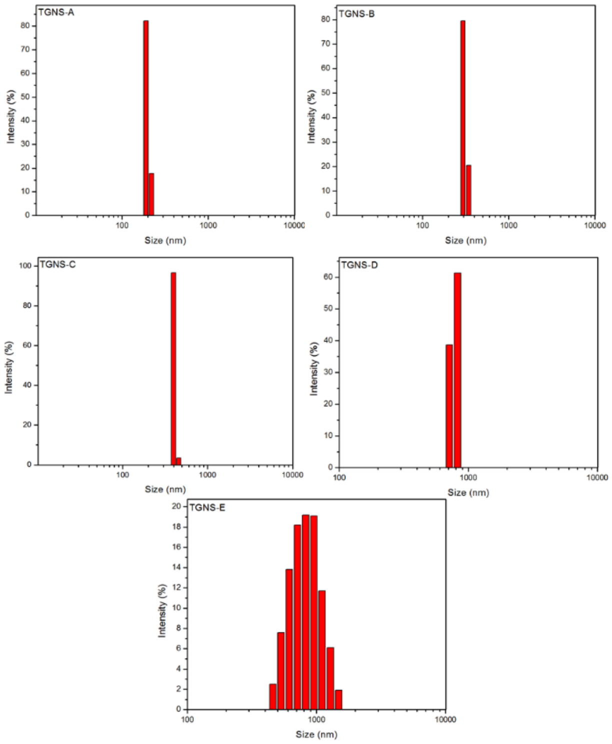

Fig.S1. Dynamic light scattering (DLS) images of all TGNS samples from A to E

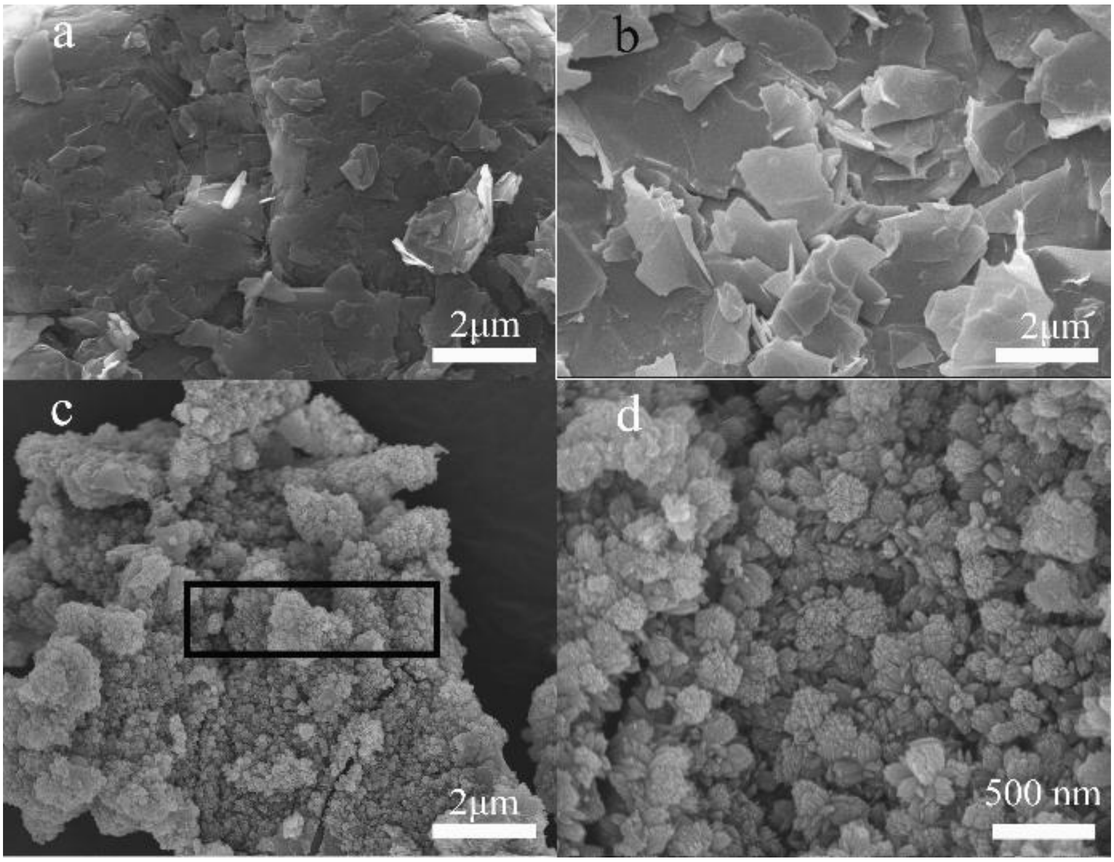

Fig.S2. SEM images of the (a) Graphite, (b) GNS, FESEM images of the (c, d) TGNS-D sample

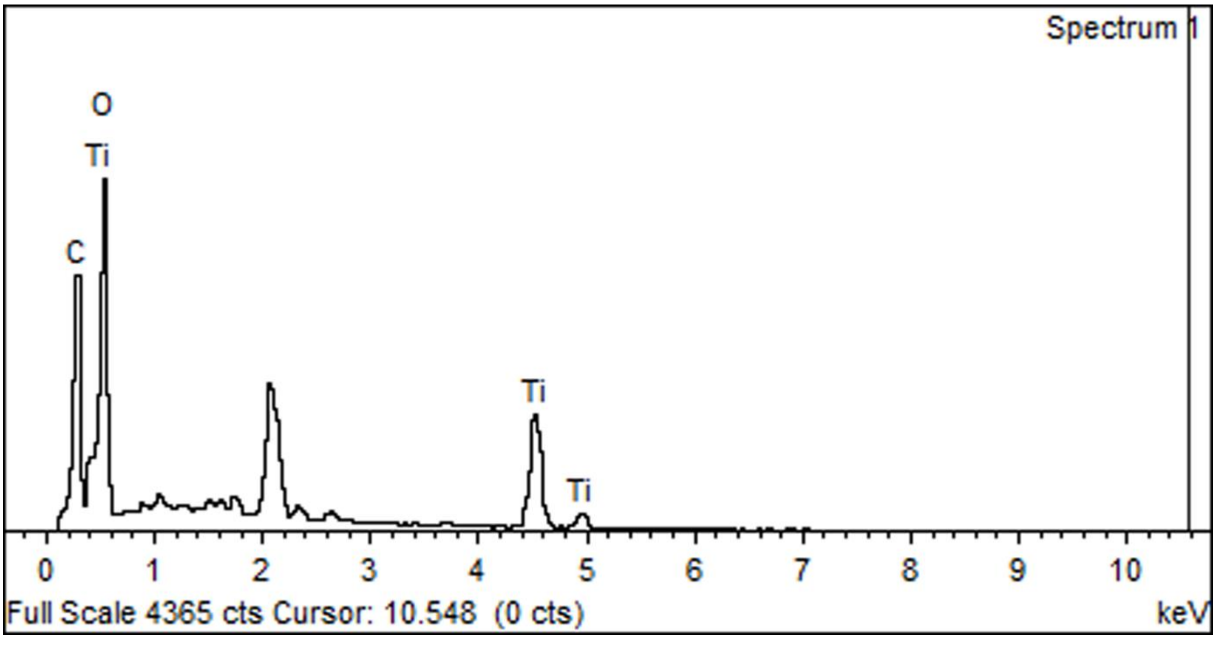

Fig.S3.The energy Dispersive Spectrometer (EDS) analysis of the black rectangular region in Fig.S2c

Sample	Sample Wt (g)	Surface area (m2)	Specific Surface area (m2·g-1)
TGNS-A	0.035	7.09	202.57
TGNS-B	0.063	12.50	198.41
TGNS-C	0.023	5.51	239.57
TGNS-D	0.018	6.07	337.22
TGNS-E	0.026	5.05	194.23
Graphite	0.061	0.31	5.08
P25	0.062	4.78	77.10

Table.S1. The specific surface area calculated by Brunauer-Emmett-Teller (BET) equation are results from A to E which correspondingly represent the TGNS samples from A to E. graphite and P25

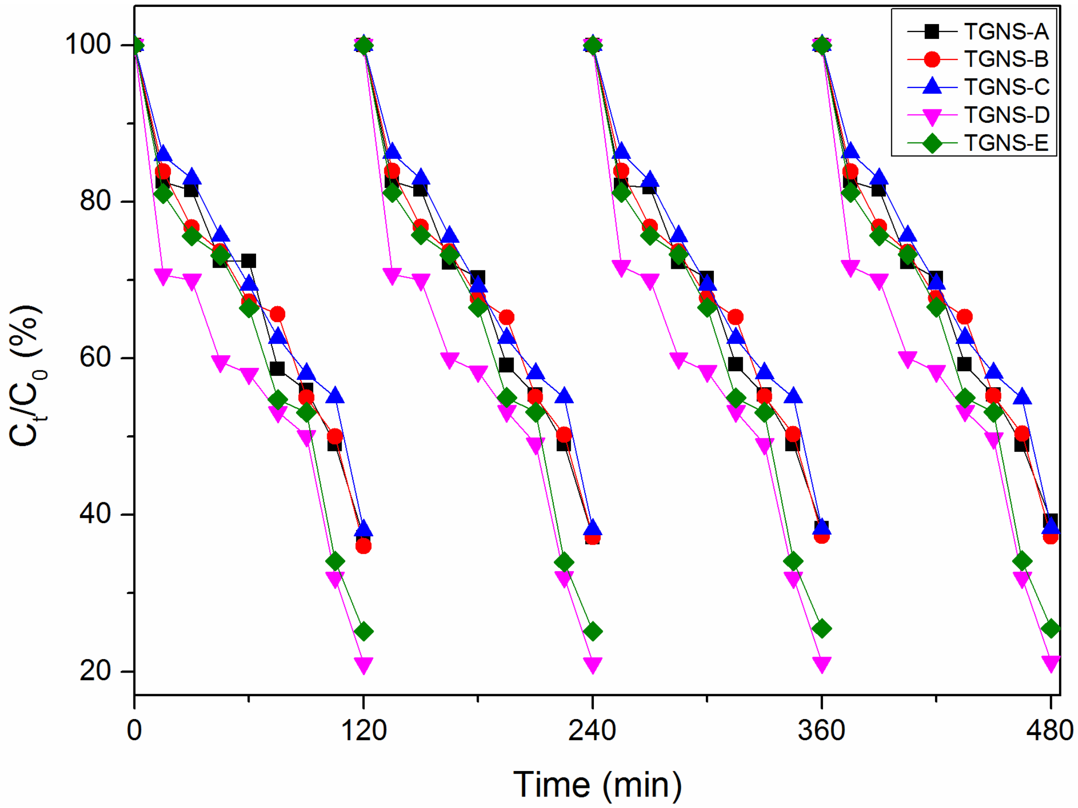

Fig.S4. Photocatalytic activity of all TGNS samples for MO degradation with 4 times of cycling uses.

UV-vis diffuse-reflectance spectroscopy

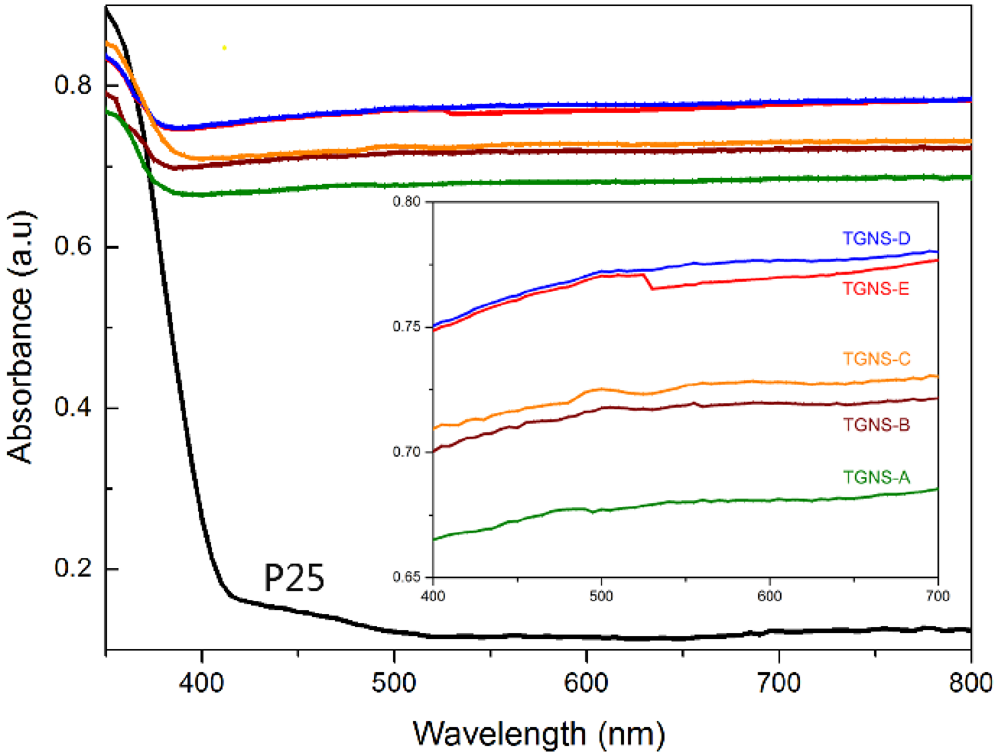

Fig.S5 UV-vis diffuse-reflectance spectroscopy of all TGNS samples and P25, the inset picture is the amplificatory image of the curves of the area in the range of 400-700 nm of all TGNS samples

From the UV-vis diffuse-reflectance spectroscopy, all TGNS samples show obvious and much better visible light absorption than bare P25. It is noteworthy that the sequence of the level of visible light absorption of TGNS samples is basically consistent with that of the level of the photocatalytic performance of them. Due to the incorporation of GNS, the visible light absorption increases significantly compared to bare P25. But the visible light absorption is not directly proportional to the amount of the addition of GNS, since the visible light absorption is also determined by the full combination of GNS and TiO_2 (the level of integrity degree of hamburger shape of TGNS samples, which can be proved from the AFM result), so there is little fluctuation between the sequence of TGNS-A to TGNS-C. The visible light absorption (400-700 nm) of the TGNS composites can be successfully proved by the UV-vis diffuse-reflectance spectroscopy.

Photoluminescence spectra

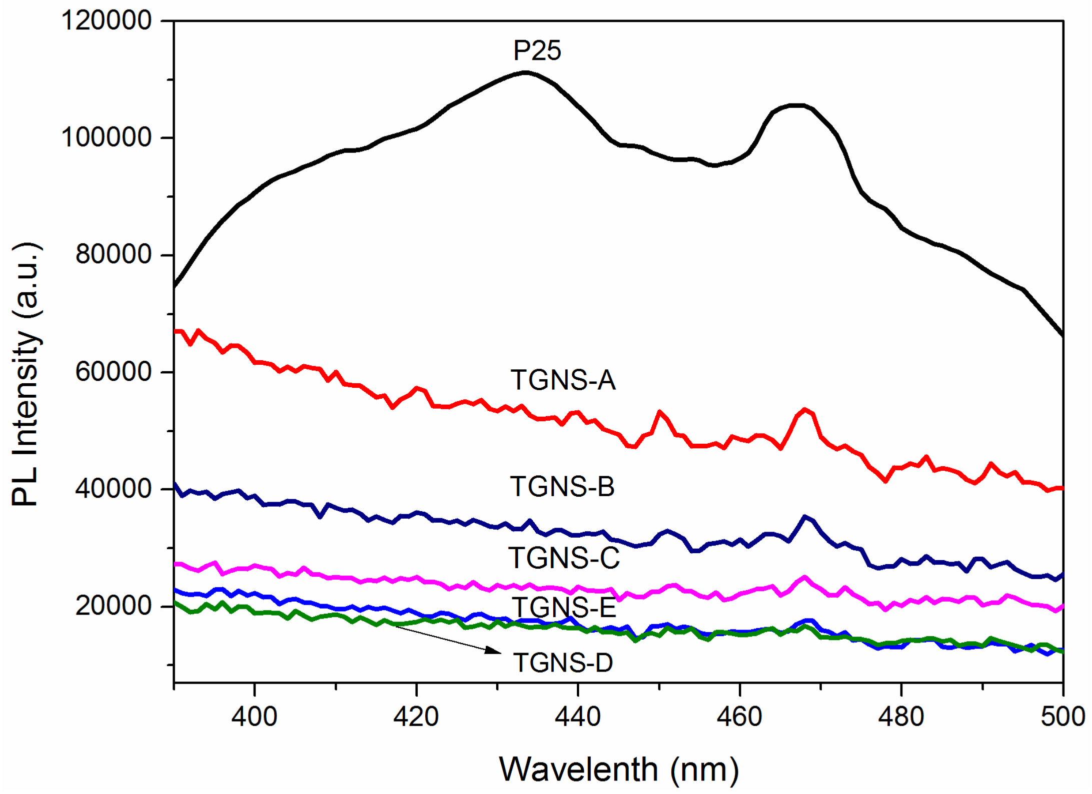

Fig.S6. Photoluminescence spectra of P25 and all TGNS composites with various reaction times (excitation wavelength is 300 nm).

The photoluminescence (PL) emission spectra have been widely applied to investigate electron/h^+ pairs of the TiO_2 related composites, such as the efficiency of charge carrier trapping, immigration and transfer [1]. Fig.S6 shows the photoluminescence spectra (PL) for bare P25 and different TGNS composites. Without the presence of other materials, electrons will transit quickly to the VB owing to the instability of excited states, resulting in the emission of fluorescence and a low catalytic activity to the pollutant. The well-resolved peaks/shoulders at 434.6 nm and 468.2 nm are attributed to the radiative recombination of photogenerated electrons and h^+ [2]. Obviously, the intensity of PL signals for the TGNS samples are much weaker than that of pure P25. In the case of TGNS composites, a heterojunction forms at the interface, where there is a space-charge separation region. The decrease of PL intensity is due to the fact that GNS has excellent conductivity, and guarantees the rapid transport and effective separation of photogenerated charge carriers. Compared to P25, the lower recombination rate of photogenerated electrons and h+ in the TGNS composites would facilitate the photocatalytic performance of TGNS composites.

References

1. H. Yamashita, Y. Ichihashi, M. Harada, G. Stewart, M. A. Fox and M. Anpo, *Journal of Catalysis*, 1996, **158**, 97-101.

2. P. Cheng, Z. Yang, H. Wang, W. Cheng, M. Chen, W. Shangguan and G. Ding, *International Journal of Hydrogen Energy*, 2012, **37**,2224-2230.