**Supplemental Information**

# A Top-Down Design for Easy Gram Scale Synthesis of Melem Nano Rectangular Prism with Improved Surface Area

Mahsa Nikookar,[a] Abdolreza Rezaeifard,[a]* Maasoumeh Jafarpour,[a]* Kirill V. Grzhegorzhevskii,[b] and Alexander A. Ostroushko[b]

[a]*Catalysis Research Laboratory, Department of Chemistry, Faculty of Science, University of Birjand, Birjand, 97175-414, Iran. E-mail: rrezaeifard@birjand.ac.ir, rrezaeifard@gmail.com.*mjafarpour@birjand.ac.ir.

[b] Institute of Natural Sciences and Mathematics, Ural Federal University named after the B.N. Yeltsin, 620002, Ekaterinburg, Russia

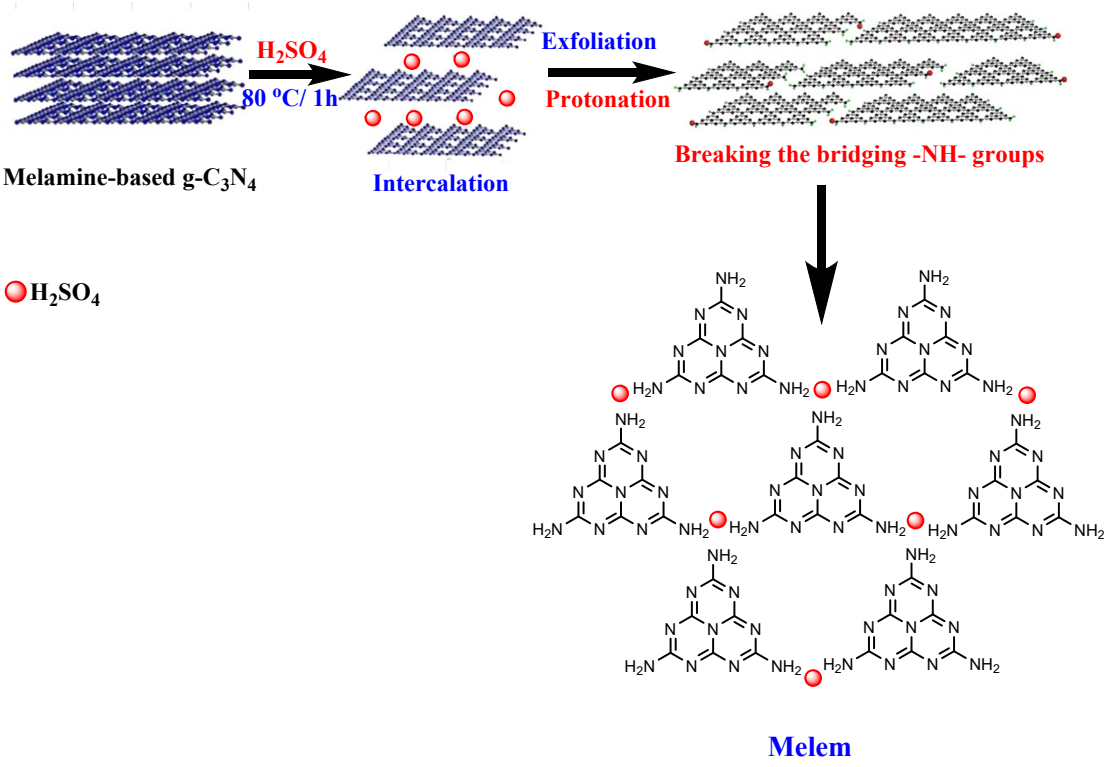
## Experimental

Analysis methods

Fourier transform infrared spectroscopy (FT-IR) spectra were recorded using a Shimadzu 800 FT-IR spectrometer using a KBr disk. NMR spectra were recorded on a Bruker Avance 300 MHz NMR spectrometer. X-ray diffraction (XRD) studies were recorded on a Bruker D8-advance X-ray diffractometer with Cu Kα (λ= 1.5406 Å) radiation. Mass spectra were recorded on an Agilent Technology (HP) with a 5973 Network Mass Selective Detector. Thermogravimetric analyses (TGA) were performed on a TGA-50 (Shimadzu) apparatus in air atmosphere with a heating rate of 10 °C/min under a nitrogen atmosphere. Photoluminescence (PL) measurements were recorded on a RF-5301DCM SHIMADZU instrument. The UV−Vis diffuse reflectance (DRS) analyses were carried out using a Avaspec-2048-TEC (Avant's spectrophotometer) in the span of 300−800 nm, and using $BaSO_4$ as a standard reference. Field Emission Scanning Electron Microscope (SEM) was performed on Zeiss sigma 300 HV. $N_2$ adsorption−desorption isotherms were performed on a Belsorp-mini II (Bel Japan) under a vacuum at 80 °C. X-ray photoelectron spectroscopy (XPS) measurements were performed using electron spectrometer K-Alpha + (Thermo Fisher Scientific, USA) with monochromatic Al Kα radiation (1486 eV). Elemental analysis was performed on an Eager 300 for EA1112 CHN Analyzer.

**Procedure for the synthesis of monomer melem from depolymerization of polymer g-$C_3N_4$**

Polymer g-$C_3N_4$ was synthesized according to a previously reported protocol [1]. Briefly, melamine (3.0 g) was calcined at 550 °C for 3 h with a heating rate of 5 °C/min under air atmosphere. The obtained yellow powder was completely grounded, then 0.2 g of the powder was added to 20 mL of concentrated $H_2SO_4$ (95-98%). The mixture was heated at 80 °C for 1 h. Then, the mixture was allowed to cool to room temperature followed by adding to a 200 mL ice-deionized water mixture. The resulting white sediment was filtered by centrifugation, washed with deionized water and ethanol several times (5 × 40 mL) to neutralize the mixture, and the white powder was dried under air (yield>95%). For further purification, it can be dissolved in dilute acid and precipitated by neutralization,[2] or dissolved in DMSO and precipitated by diethyl ether (EA%; C:32.71, H:2.93, N:63.0, S:0.41%). A 5 fold semi-scaled up procedure using 1.0 g g-$C_3N_4$ polymer led to the isolation of the related pure monomer melem in 95% yield within 1 h.




**Scheme S1.** The suggested mechanism for the acidic depolymerization of melamine-based g-$C_3N_4$ to monomer melem.

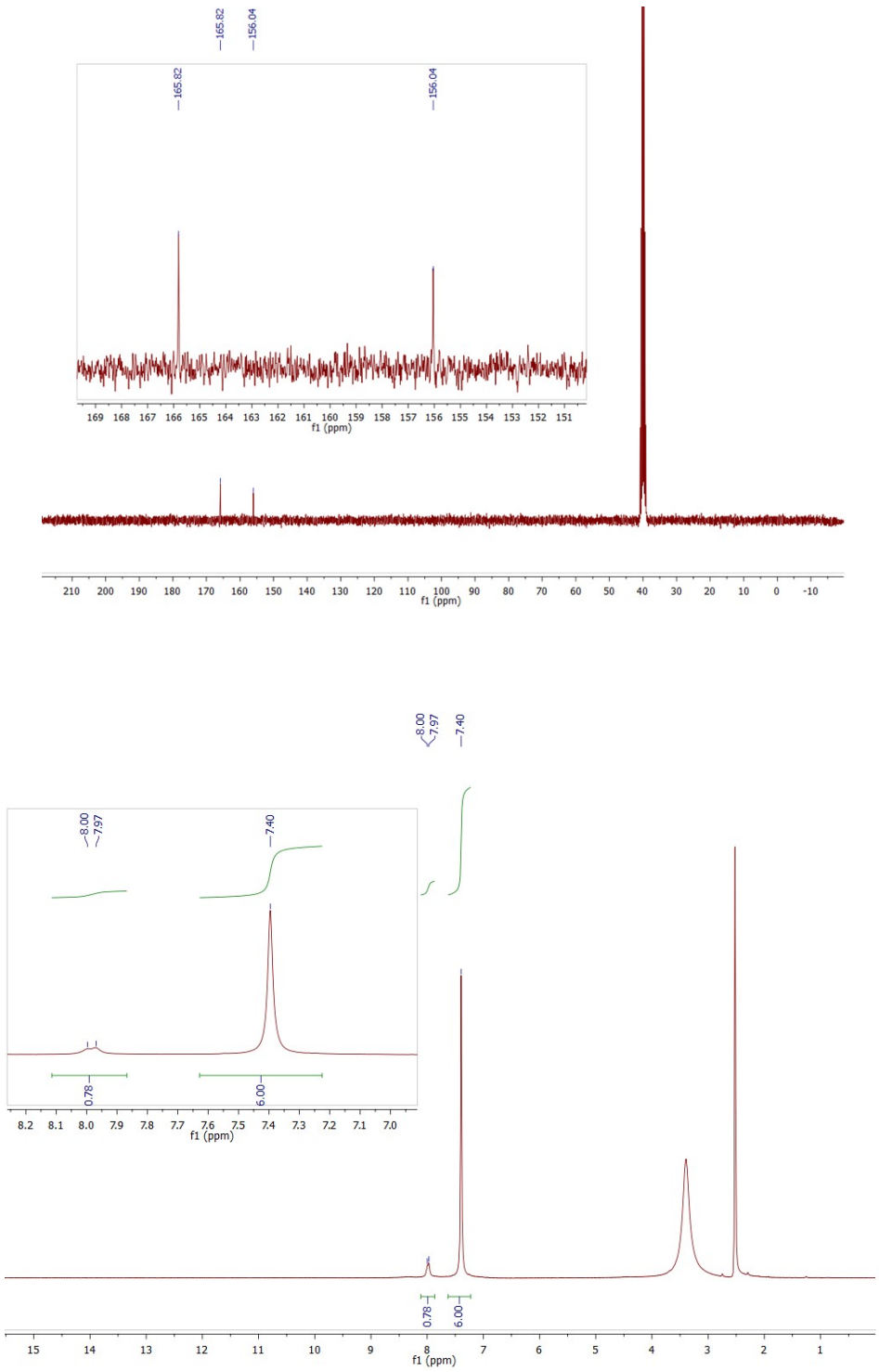



**Figure S1.** $^{13}C$ NMR (top) and $^{1}H$ NMR spectrum (down) of the as-synthesized melem in DMSO before further purification. The sample is only dissolved in DMSO with very limited solubility and for $^{13}C$ NMR it has to stay 3-4 days in DMSO-$d^6$.

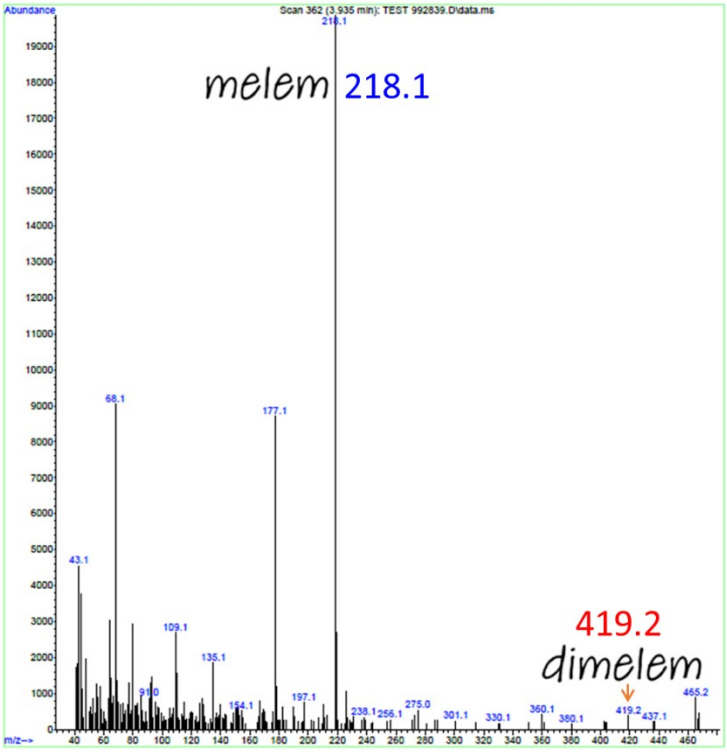



Figure S2. MS spectra of the as-synthesized melem before further purification

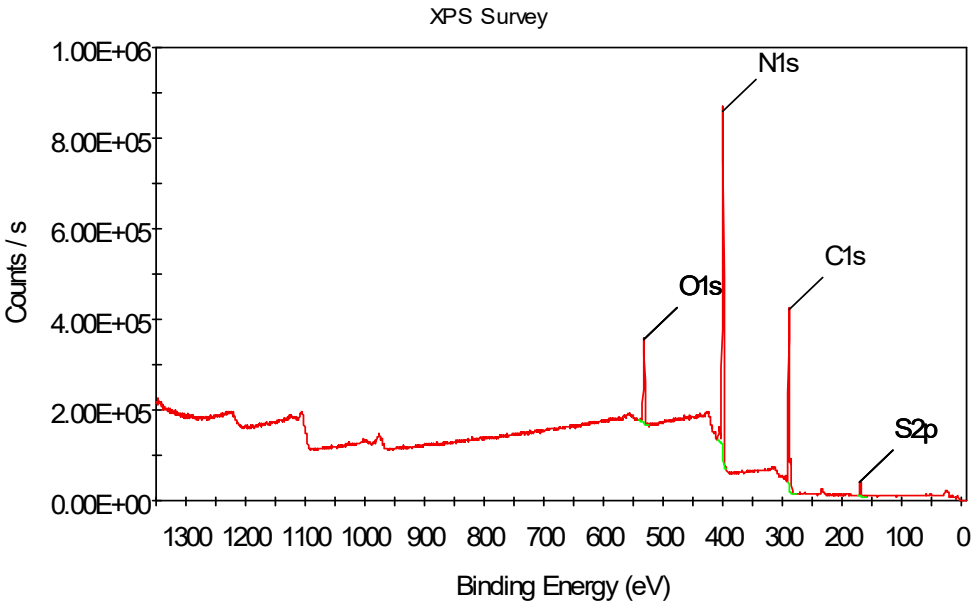



Figure S3. XPS survey of the as-synthesized melem before further purification

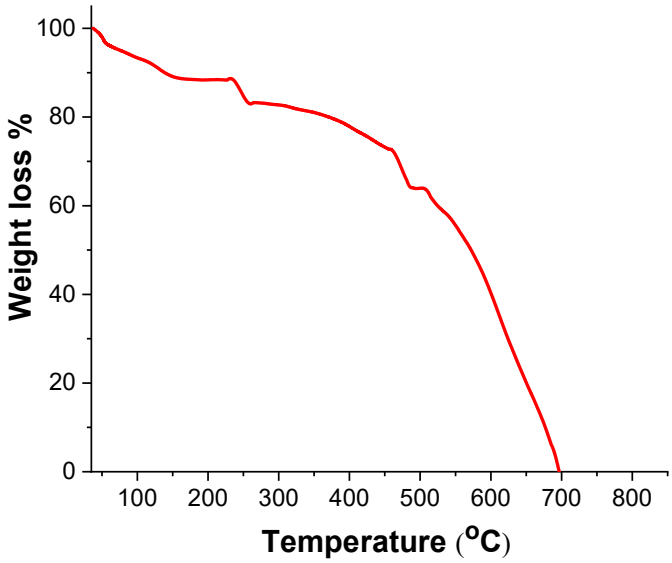



Figure S4. TGA curve of the as-synthesized melem under a nitrogen atmosphere before further purification

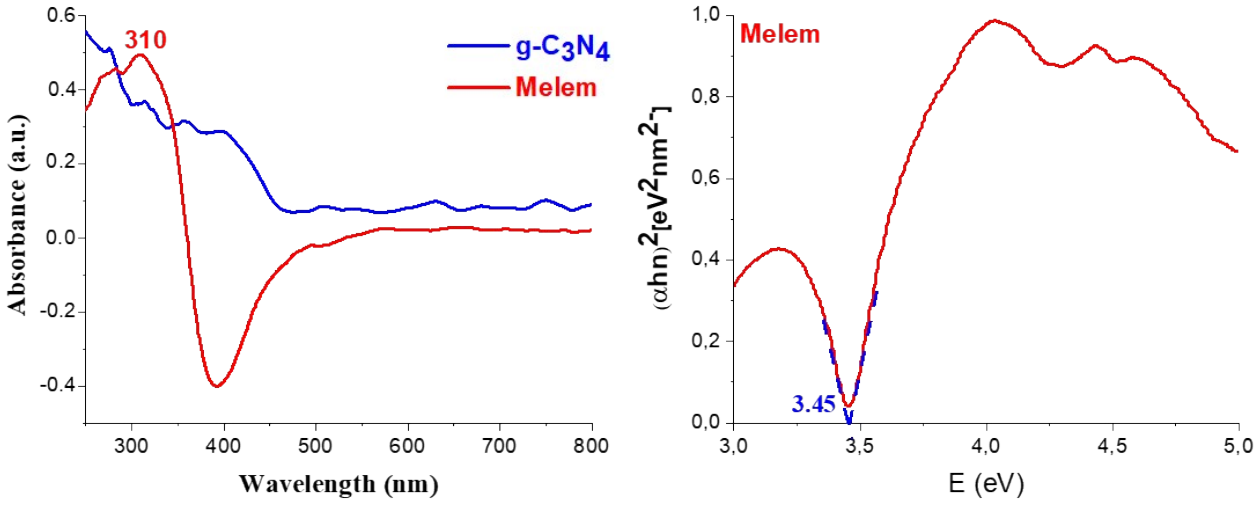



Figure S5. DRS UV-Vis spectra of the as-synthesized g-$C_3N_4$ and melem (left) and tauc's plot of melem (right). The recorded negative absorption of melem below 400 nm comes from the stronger adsorption of the $BaSiO_4$ reference.[3]

## Reference:


1. Liu, S.; Wang, S.; Jiang, Y.; Zhao, Z.; Jiang, G.; Sun, Z. Synthesis of $Fe_2O_3$ loaded porous g-$C_3N_4$ photocatalyst for photocatalytic reduction of dinitrogen to ammonia. *Chem. Eng. J.* 2019, **373**, 572-579.
2. A. I. Finkelshtein and N. V. Spiridonova, Russ. Chem. Rev., 1964, **33**, 400–405.
3. Liu, N.; Li, T.; Zhao, Z.; Liu, J.; Luo, X.; Yuan, X.; Luo, K.; He, J.; Yu, D.; Zhao Y.; *ACS Omega* 2020, **5**, 12557–12567.