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Supporting Information

Assembly of Ring-Shaped Phosphorus within Carbon Nanotube Nanoreactors

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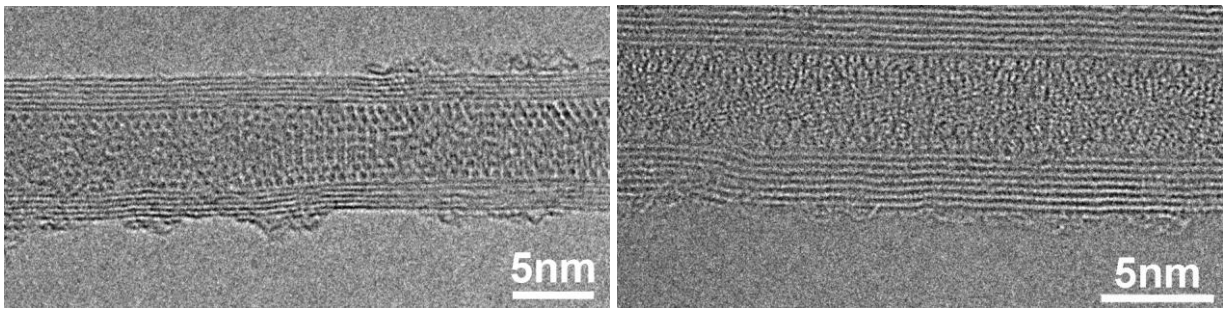
Experimental dails:

Synthesis: MWCNTs were produced by chemical vapor deposition (CVD) using Co/Fe-Al_2O_3 as catalysts, with ethylene as carbon source and purified. The purified MWCNTs were heated at 500°C for 30 min under an air atmosphere to open the caps. The open-ended MWCNTs were degassed for one day with interval flame heating and then sealed in the presence of extra red phosphorus (Aladdin, 99.999% metals basis) under a vacuum of 10^{-5} Pa in an H-shaped Pyrex tube. The H-shaped Pyrex tube was then heated to 500°C for 48 h at a heating rate of 1°C/min and then cooled down in oven or under a temperature gradient condition (one end about 50~100°C lower than the other end where the MWCNTs were present).

Raman: Raman spectroscopy was taken in a back-scattering geometry using a single monochromator with a microscope (Reinishaw inVia), which was equipped with a CCD array detector (1024 × 256 pixels, cooled to -70ºC) and an edge filter. The Raman spectra were recorded at a power of 0.49-7.0 mW for 100-500 s with excitation lasers at 514 and 633 nm. The spectral resolution and reproducibility was determined to be better than 0.1 cm^{-1}.

FTIR: ATR-FTIR was performed by a BRUKER/TENSOR27 spectrometer using Single Reflection ATR by PIKE Technologies. The spectra were obtained with a spectral resolution of 4 cm^{-1} for 1000 scans.



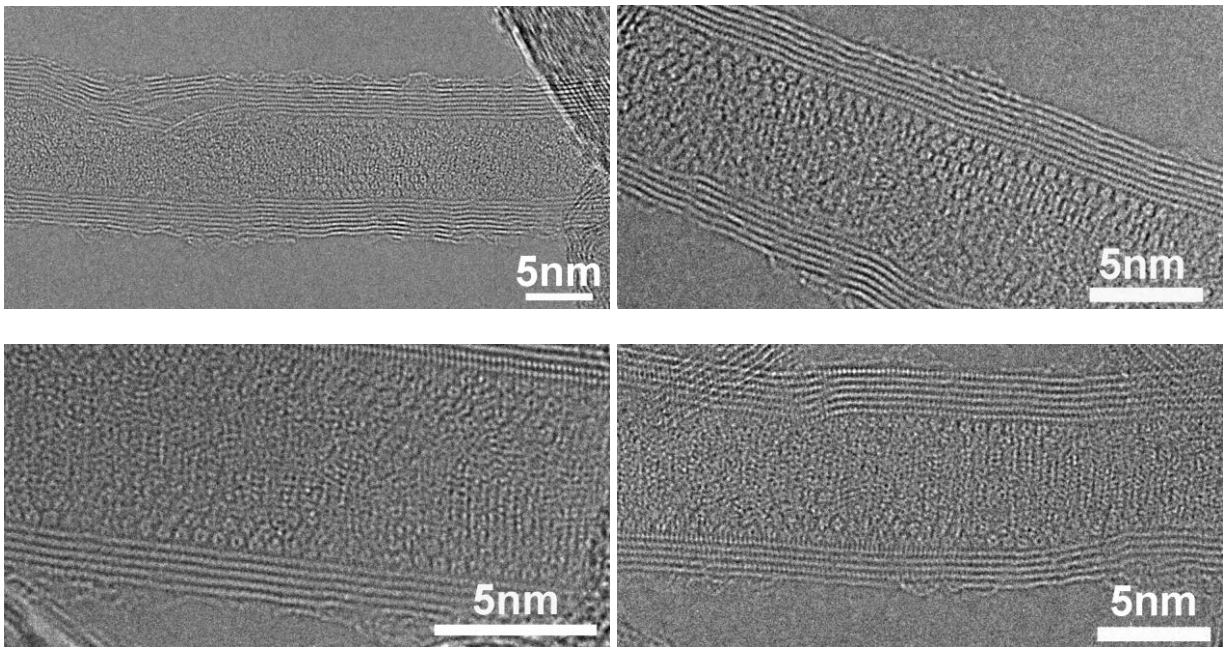


Figure S1. HR-TEM images of *r*-P@MWCNTs (FEI Titan G2, accelerating voltage:300 kV).

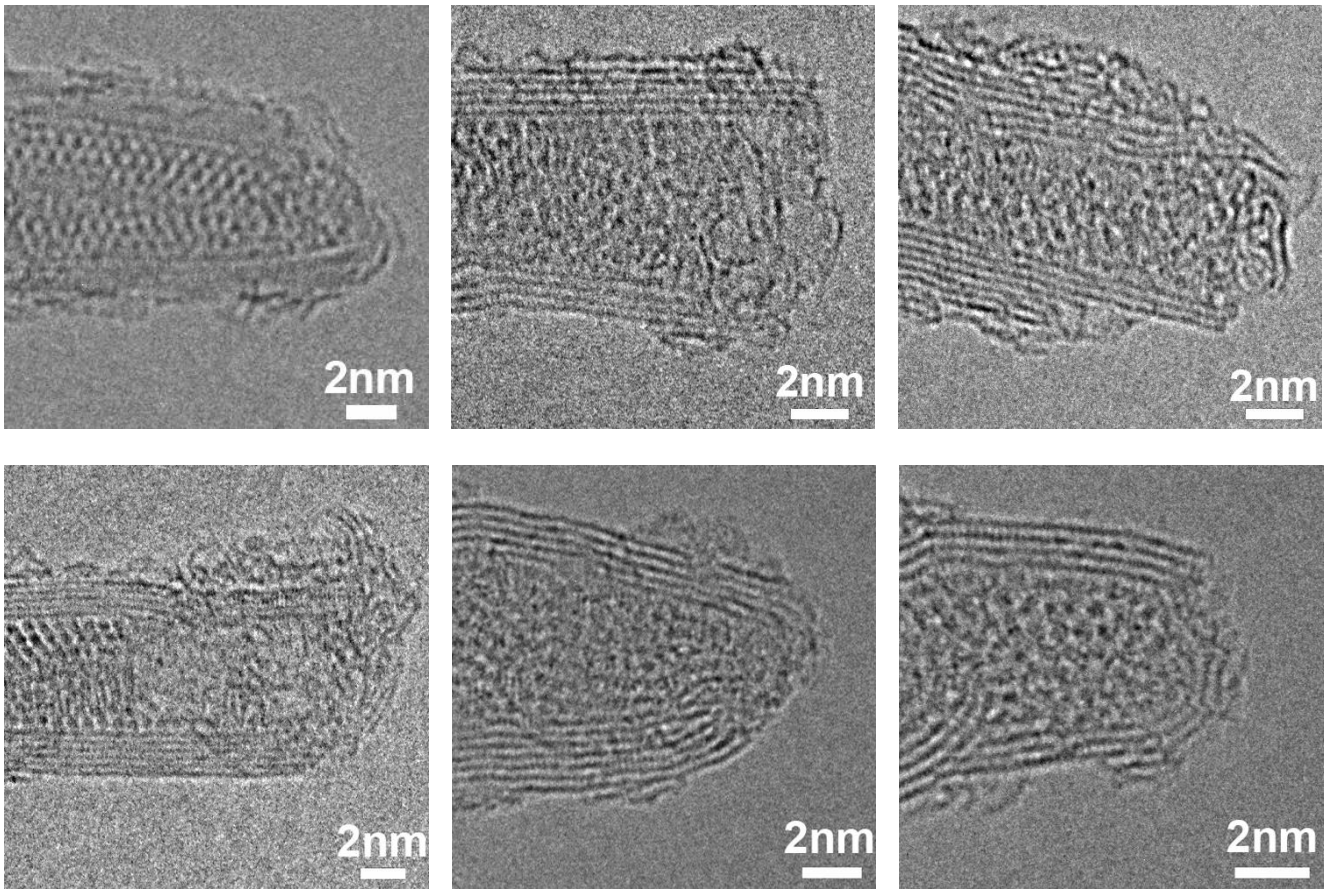
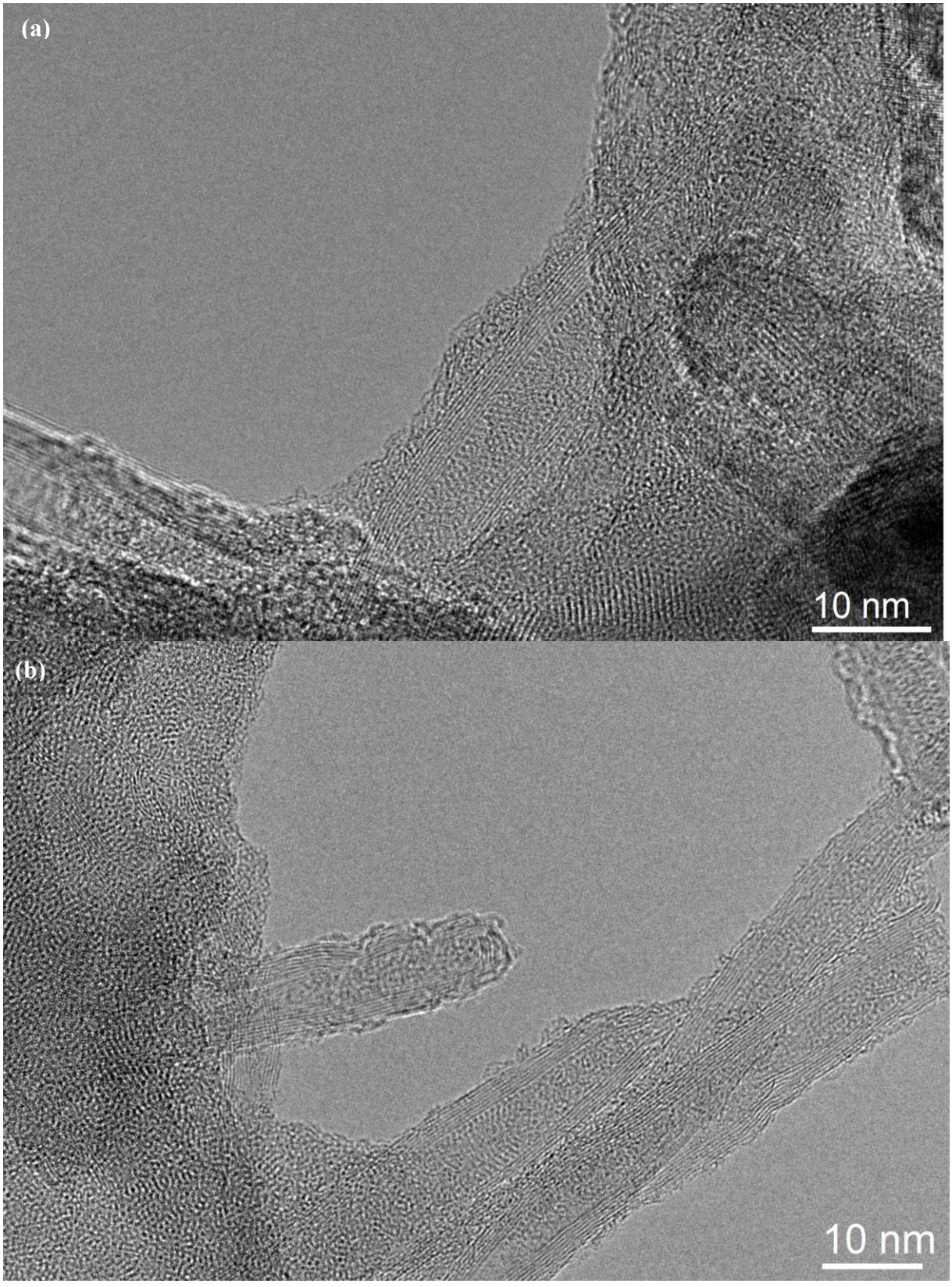


Figure S2. HR-TEM images of the end of *r*-P@MWCNTs (FEI Titan G2, accelerating voltage:300 kV).


(a)
10 nm
(b)
10 nm

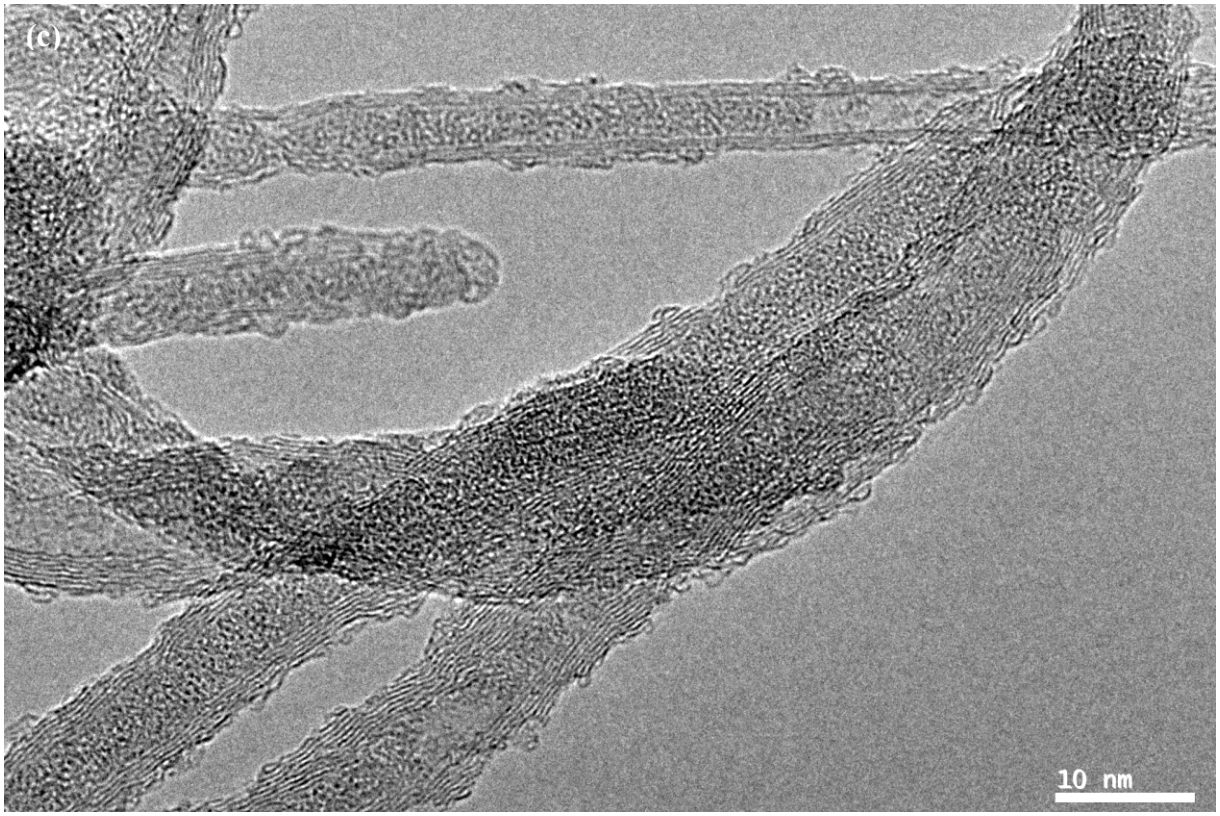
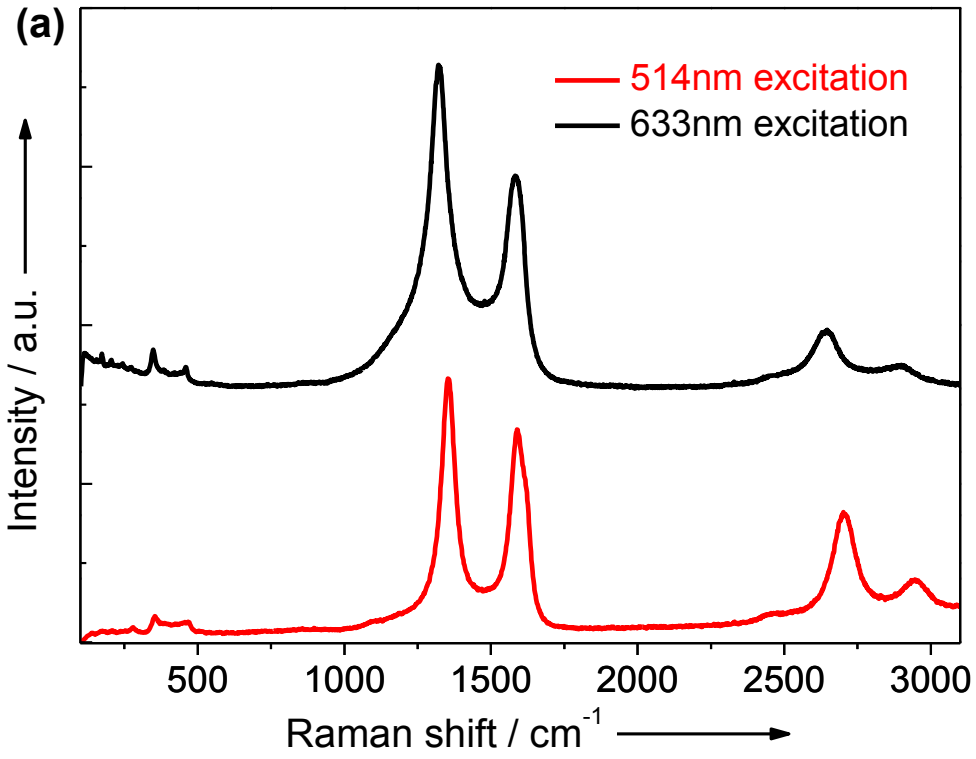


Figure S3. HR-TEM images of *r*-P@MWCNTs (a) before and (b) after washing, (c) after temperature gradient treatments.



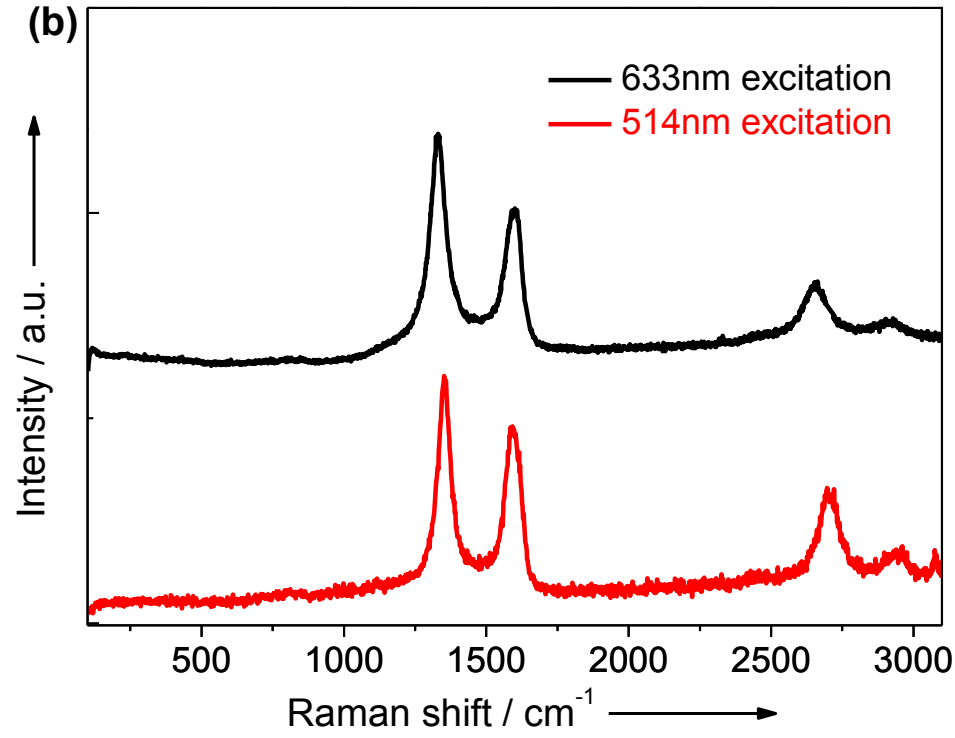


Figure S4. Raman spectrum of *r*-P@MWCNTs in a) air and b) Ar atmosphere

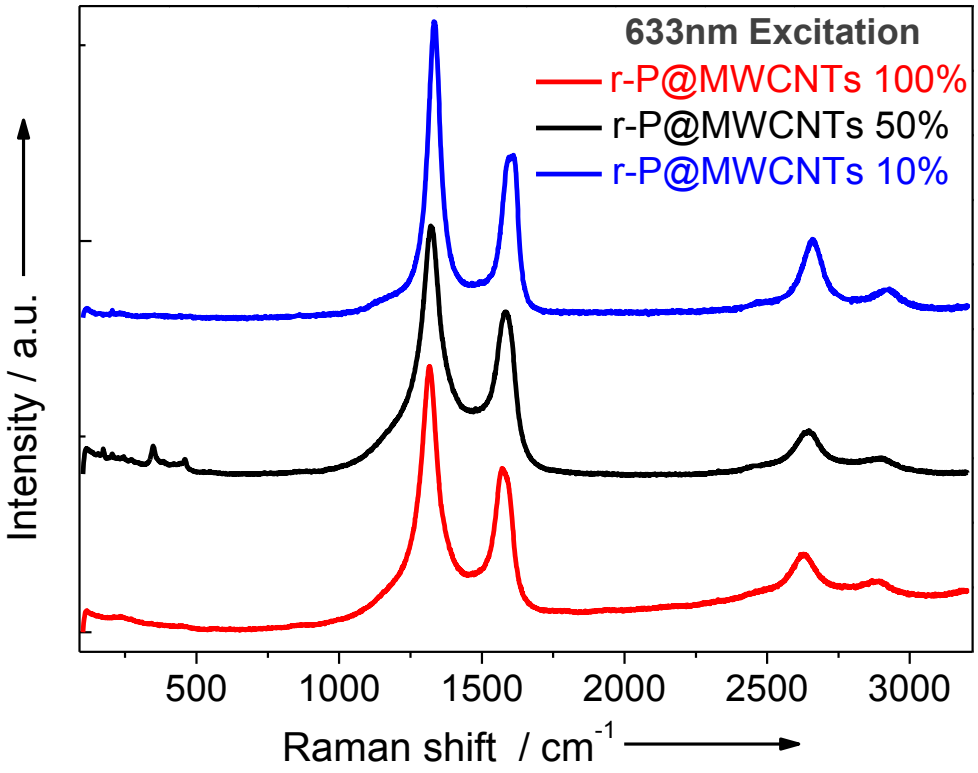


Figure S5. Raman spectrum of *r*-P@MWCNTs with different laser power.

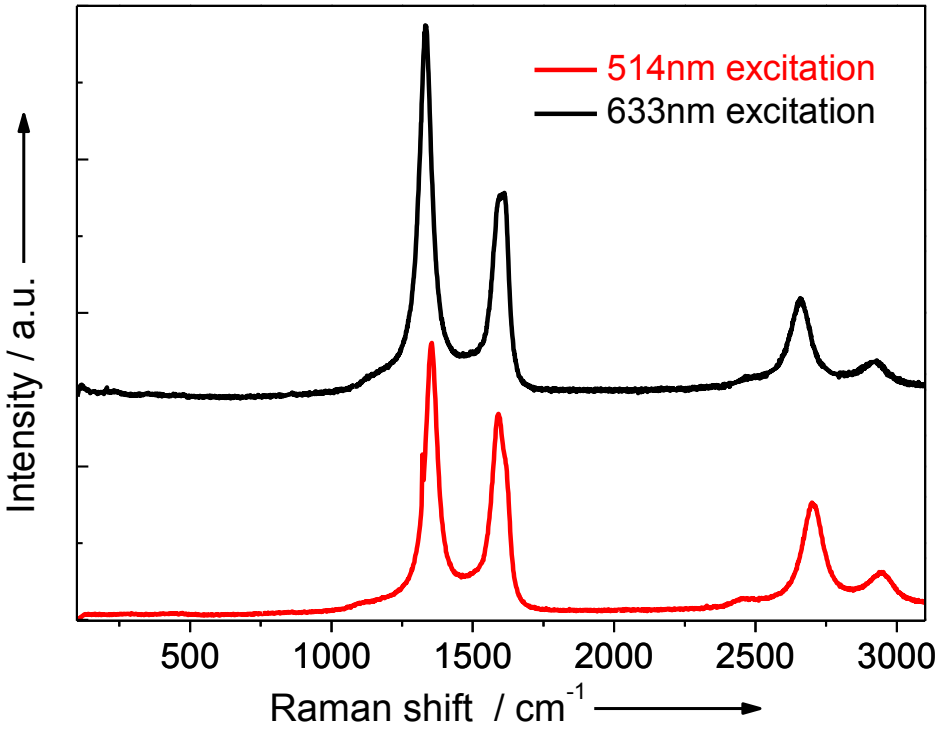


Figure S6. Raman spectrum of TGC-*r*-P@MWCNTs under ambient conditions