

# Synthesis of covalent boroxine frameworks via polycondensation of tetrahydroxydiboron

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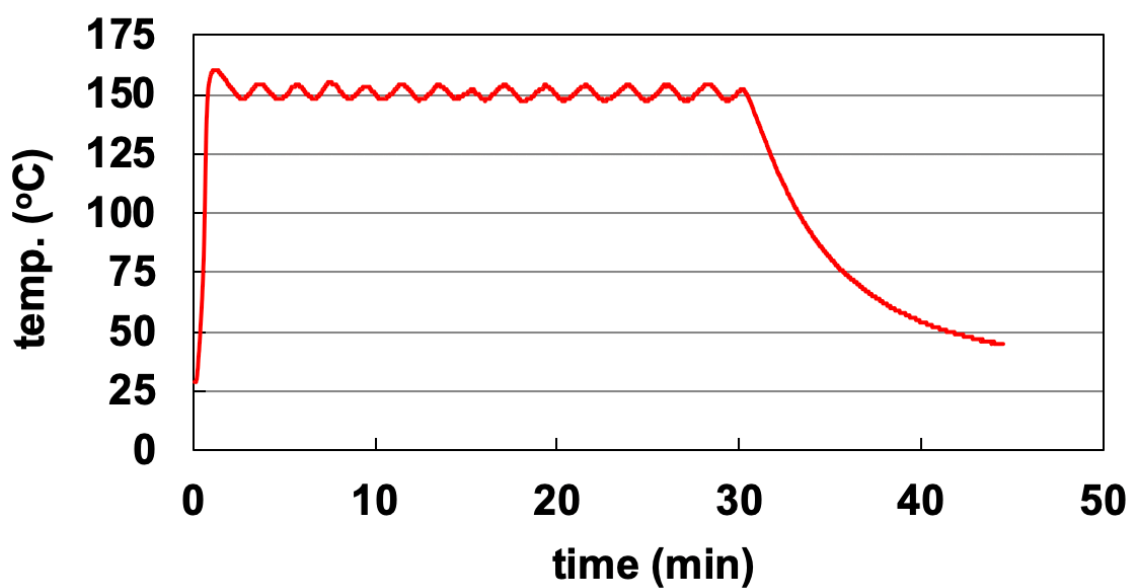


Figure S1. Plots of temperature on time.

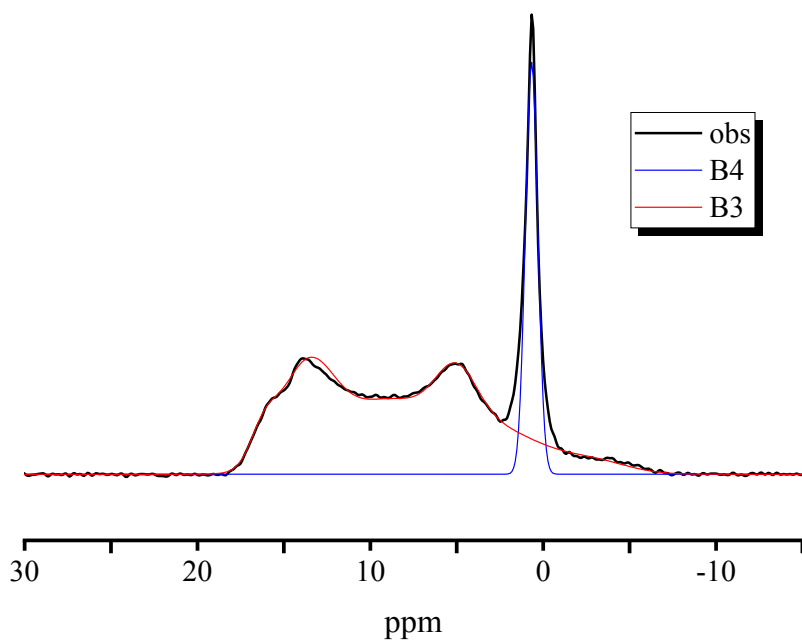


Figure S2. Deconvolution of  $^{11}\text{B}$  MAS NMR spectrum of CBOF2.

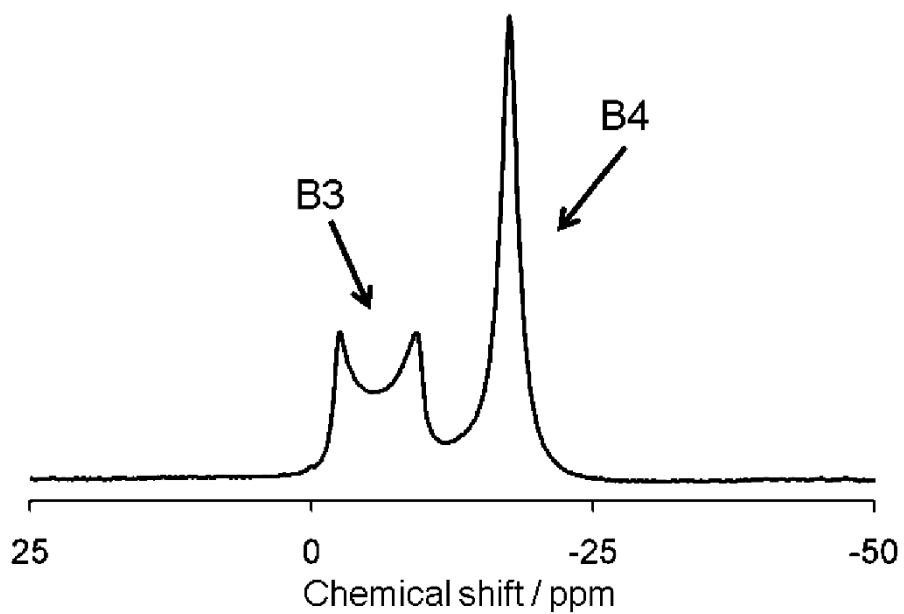


Figure S3.  $^{11}\text{B}$  MAS NMR spectrum of borax measured at 11.7 T. Original source by K. Yamada<sup>7</sup>, copyright with permission from American Chemical Society (2011).

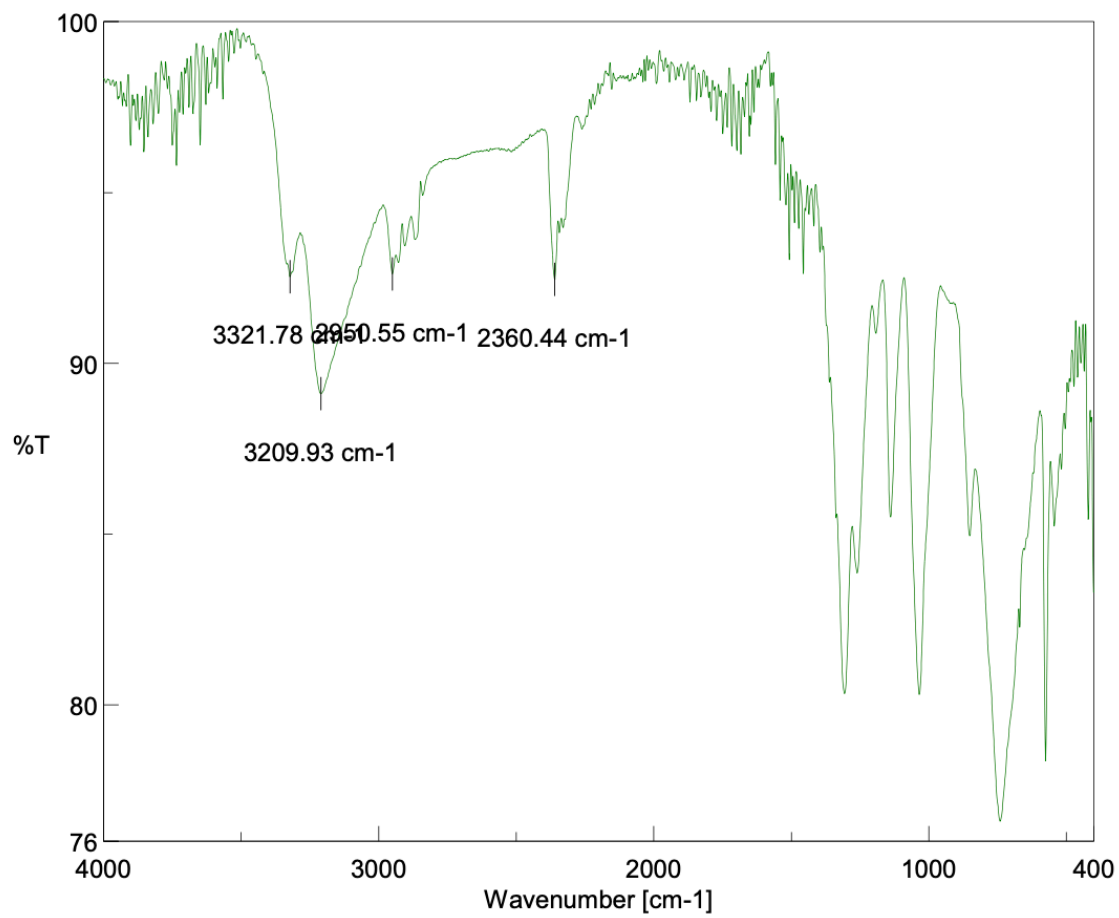


Figure S4. IR spectrum of **THB**.

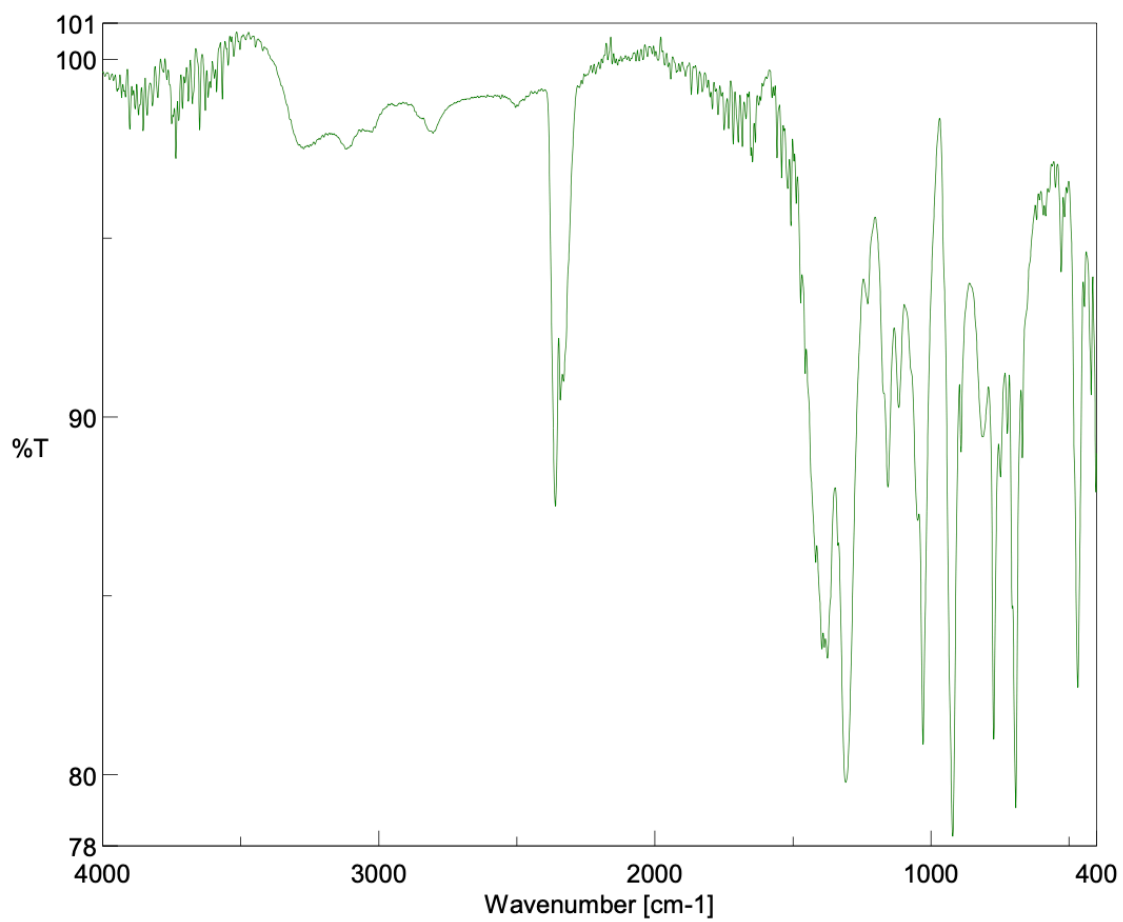


Figure S5. IR spectrum of **CBOF1**.

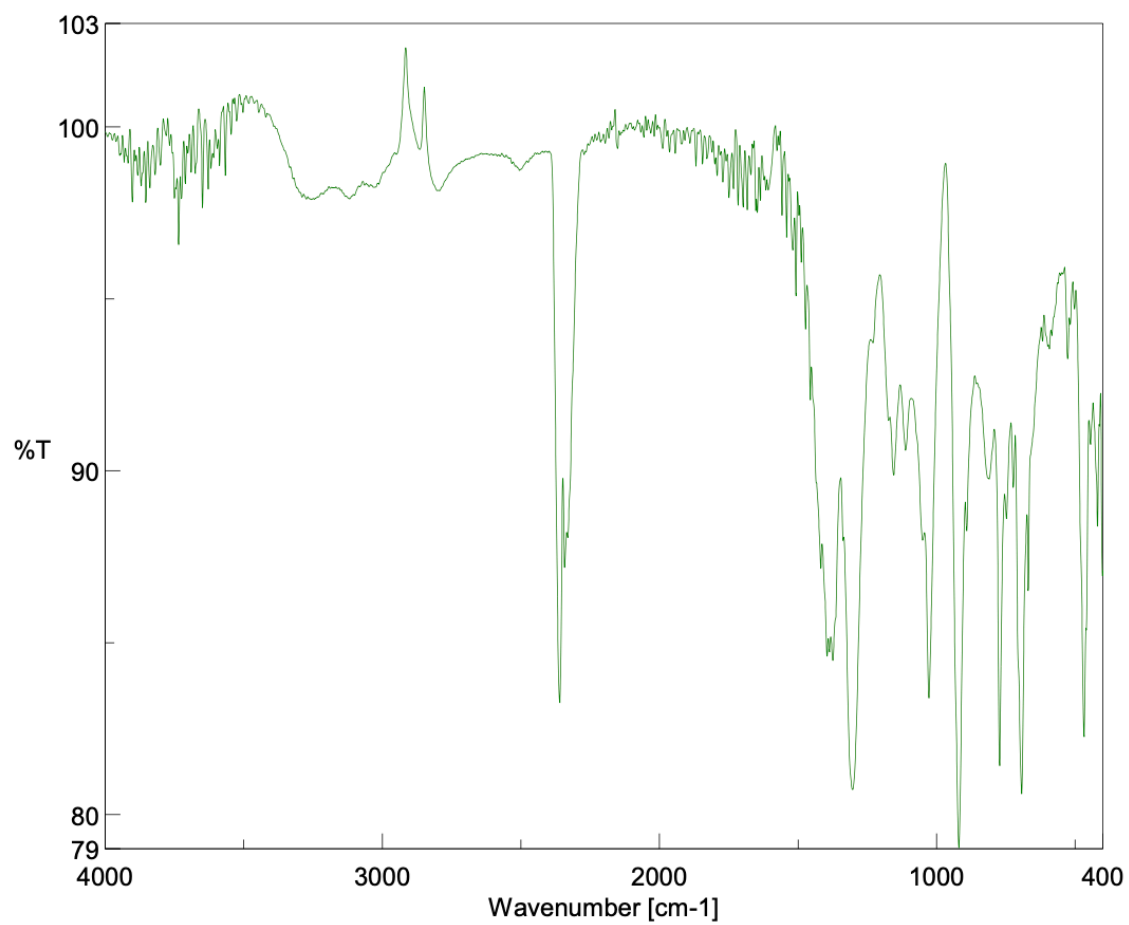


Figure S6. IR spectrum of **CBOF2**.

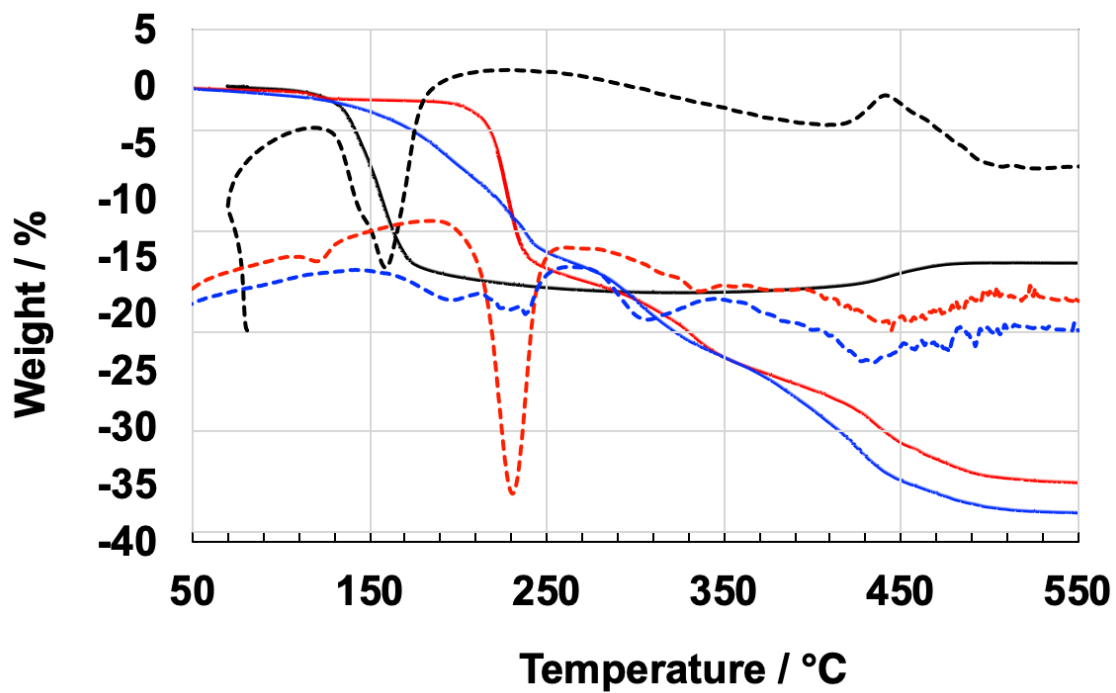


Figure S7. TG (solid line) and DTA (dotted line) traces of **THB** (black), **CBOF1** (red), and **CBOF2** (blue).

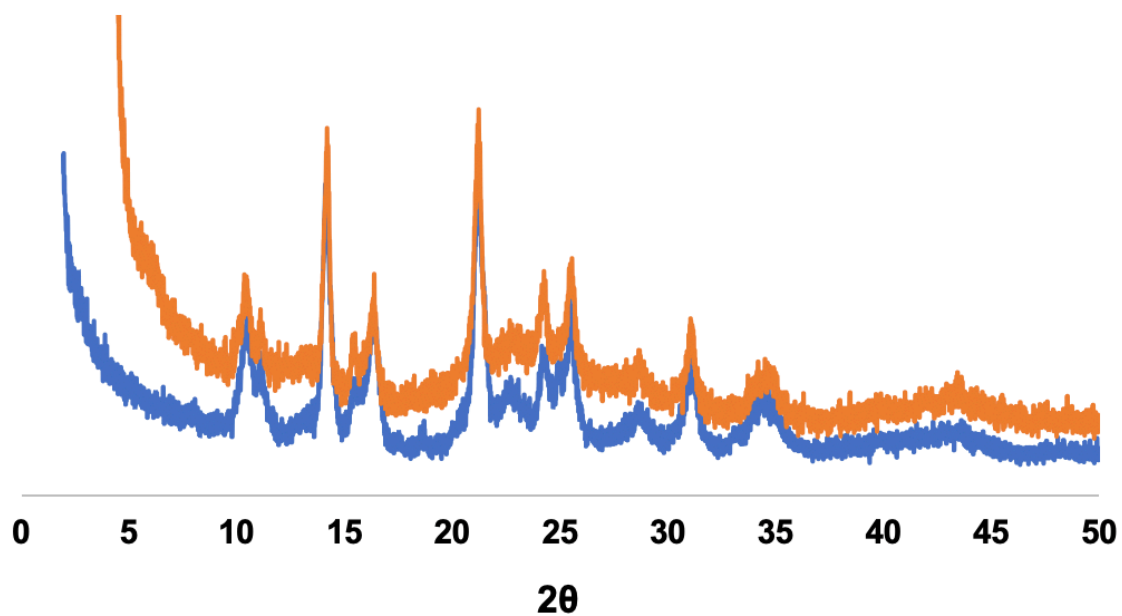


Figure S8. Powder XRD patterns of before (orange) and after (blue) 240°C heating.

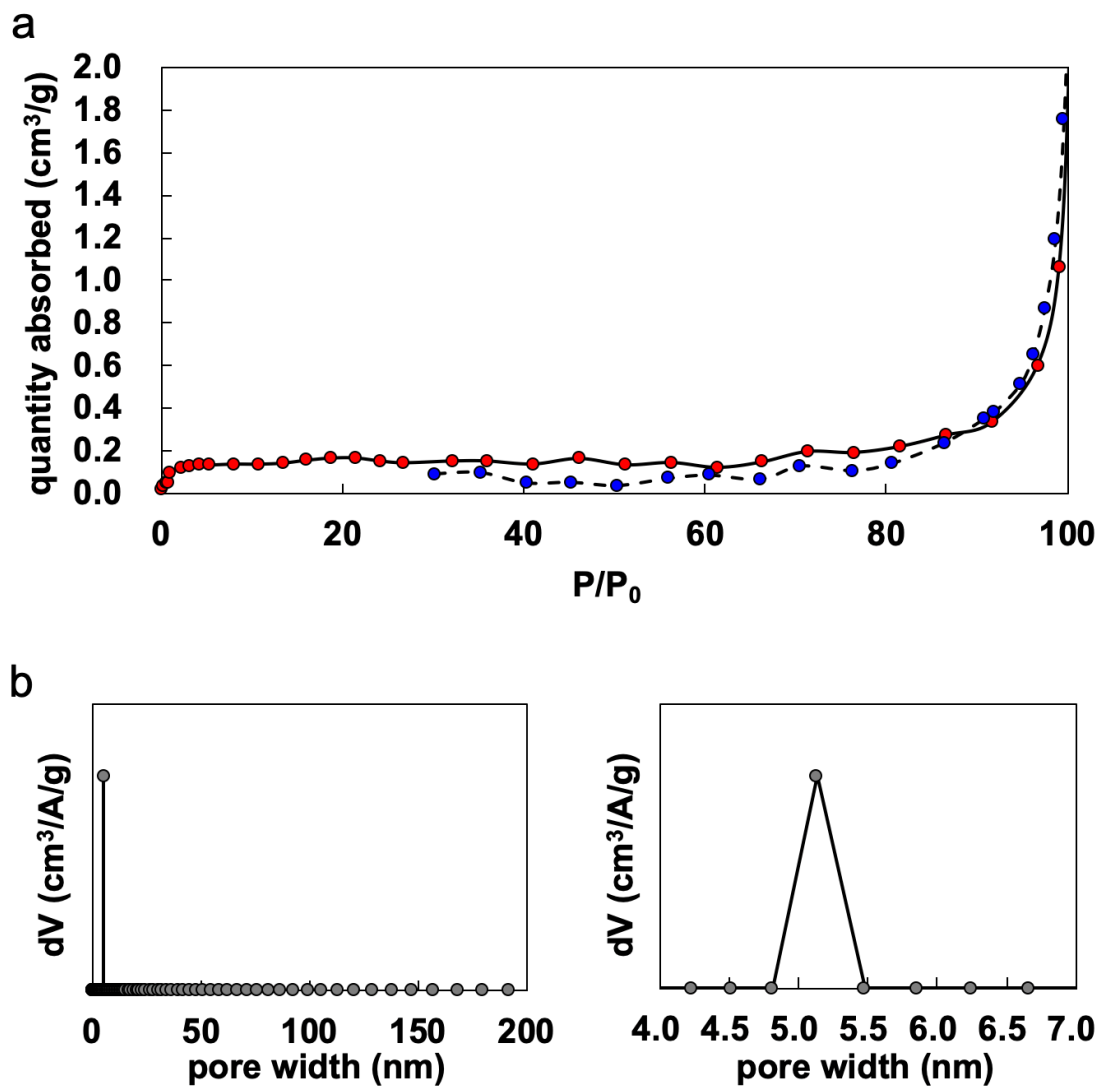


Figure S9. (a) Nitrogen adsorption isotherms of **CBOF2** measured at 77 K (the adsorption and desorption branches are labeled with red and blue symbols, respectively). (b) Pore size distribution curves of calculated by NLDFT method.