

Supplementary Materials for

Preparation of POSS-modified phenolic aerogel/quartz fiber composites: low density, high strength, great thermal insulation and ablation resistance

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S1 Equation parameter fitting

Based on the Kissinger (1), Ozawa (2), and Crane (3) equations, the apparent activation energy (E_a) and reaction series of the curing reactions were calculated as follows:

$$\ln\left(\frac{\beta}{T_p^2}\right) = \ln\left(\frac{AR}{E_a}\right) - \frac{E_a}{R} \cdot \frac{1}{T_p} \quad (1)$$

$$\ln\beta = \ln\frac{AE_a}{R} - \ln g(a) - 5.331 - 1.052 \frac{E_a}{RT_p} \quad (2)$$

$$\frac{d(\ln\beta)}{d\left(\frac{1}{T_p}\right)} = -\frac{E_a}{nR} - 2T_p \quad (3)$$

Where β is the heating rate ($^{\circ}\text{C}/\text{min}$); T_p is the peak temperature of the curing reaction. E_a is the apparent activation energy of the curing, R is the gas constant, and A is the pre-exponential factor. Based on the DSC results, a reasonable curing procedure was designed as follows: $120\text{ }^{\circ}\text{C}/2\text{ h} \rightarrow 145\text{ }^{\circ}\text{C}/3\text{ h} \rightarrow 175\text{ }^{\circ}\text{C}/2\text{ h} \rightarrow 210\text{ }^{\circ}\text{C}/3\text{ h} \rightarrow 230\text{ }^{\circ}\text{C}/1\text{ h}$ (Figure. S1).

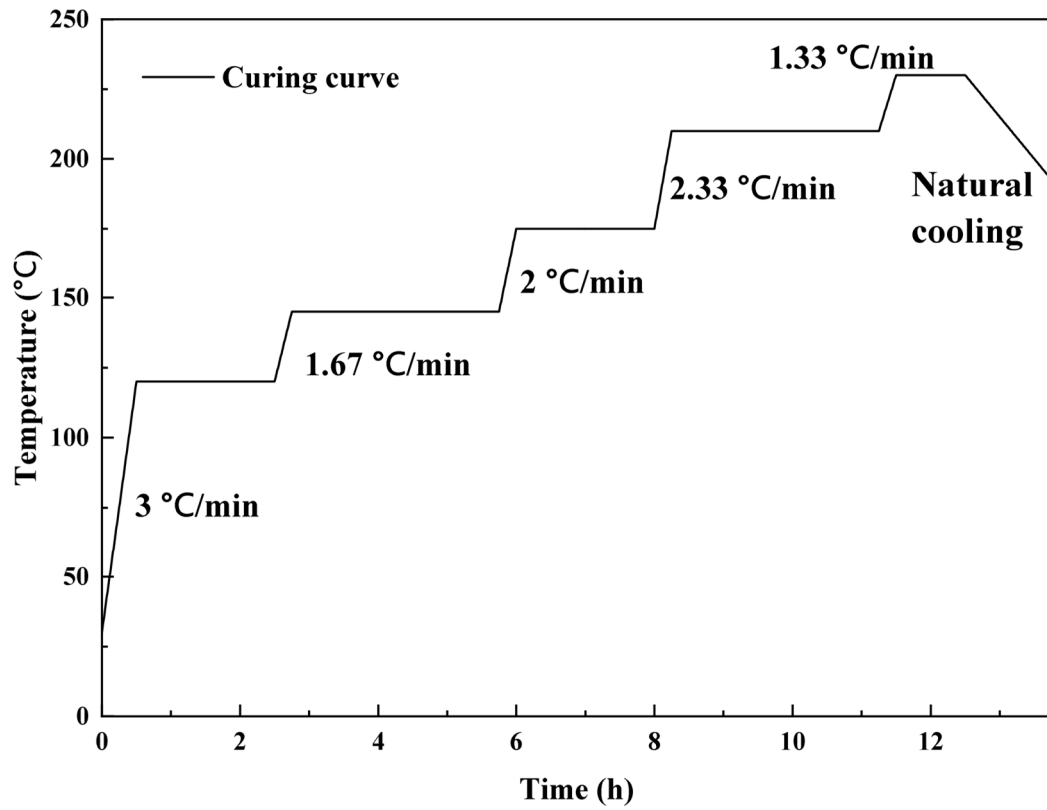


Figure. S1 Curing process

S2 SH-POSS GPC results

Molecular weight-retention time fitting equation after GPC column calibration:

$$\log M = -0.915672t + 11.7027 \quad (6)$$

Where M is the molecular weight and t is the retention time.

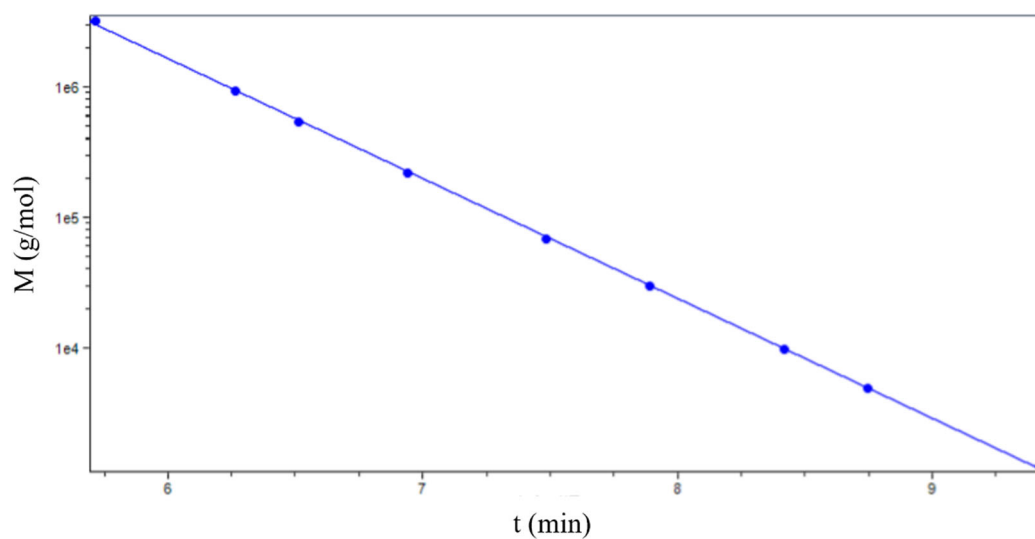


Figure. S2 GPC column calibration curve

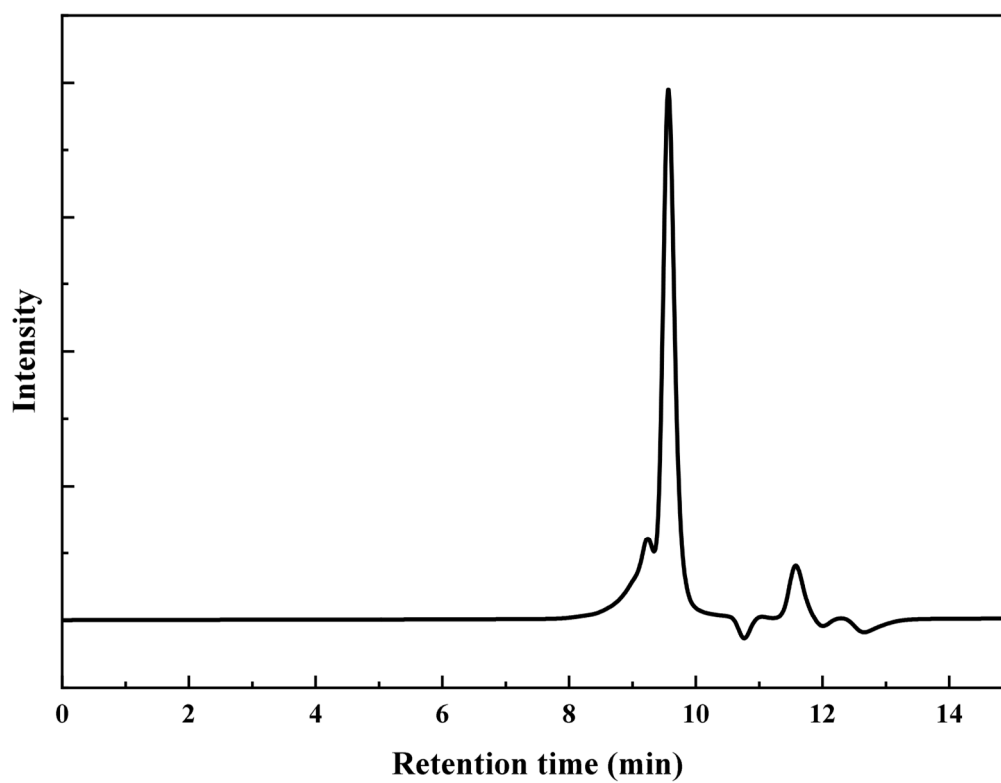


Figure. S3 GPC chromatogram

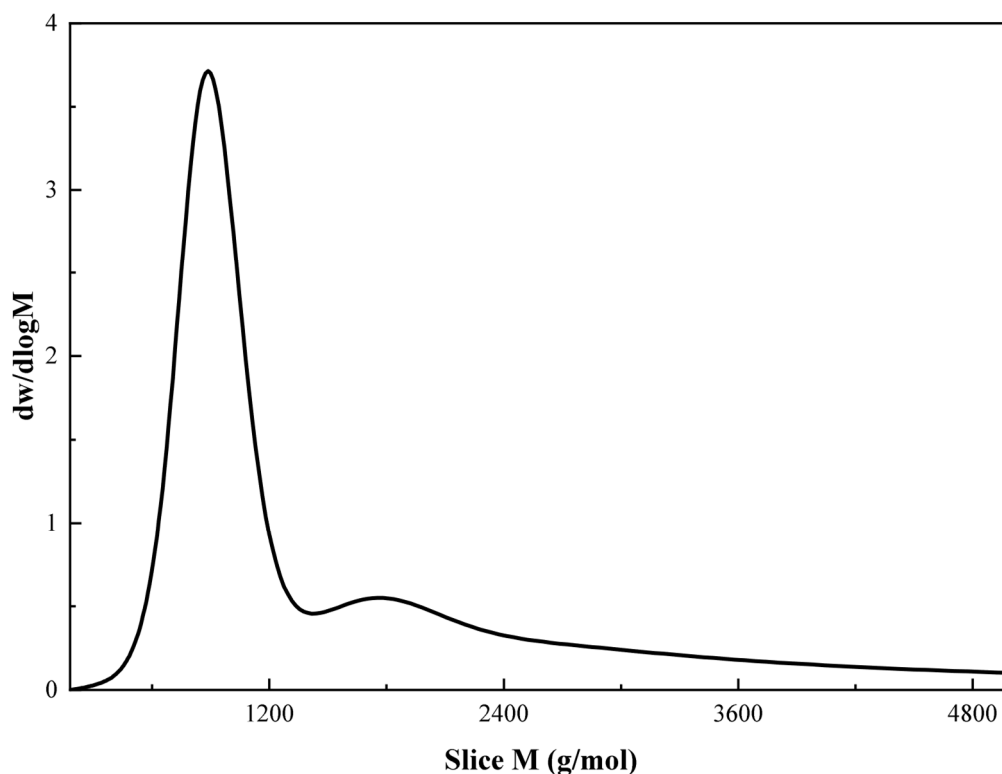


Figure. S4 Molecular weight distribution curve

Table S1 Molecular weight statistical results

M _p (g/mol)	M _n (g/mol)	M _w (g/mol)	M _z (g/mol)	M _{z+1} (g/mol)	M _η (g/mol)	Polydispersity
886	940	1183	2589	6398	1192	1.259

According to the above GPC test results (Figure. S2, S3, S4 and Table S1), the actual weight-average molecular weight of SH-POSS (1183) is close to the theoretical value (1016), which proves the successful synthesis of SH-POSS; combined with the polydispersity coefficient (1.259) and the molecular weight distribution curve, it shows that the product contains a small amount of non-T8-POSS, such as T7-POSS, T10-POSS, etc.

S3 PR and POSS-PR BET analysis

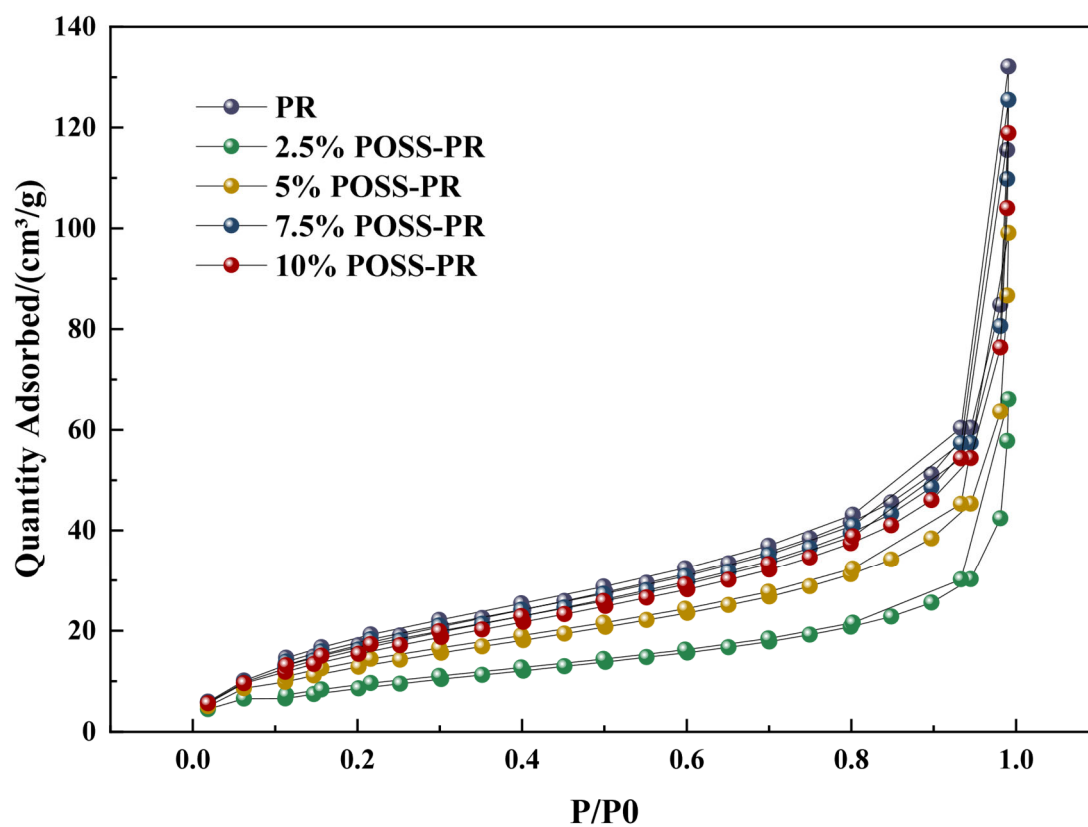


Figure. S5 BET adsorption-desorption curves

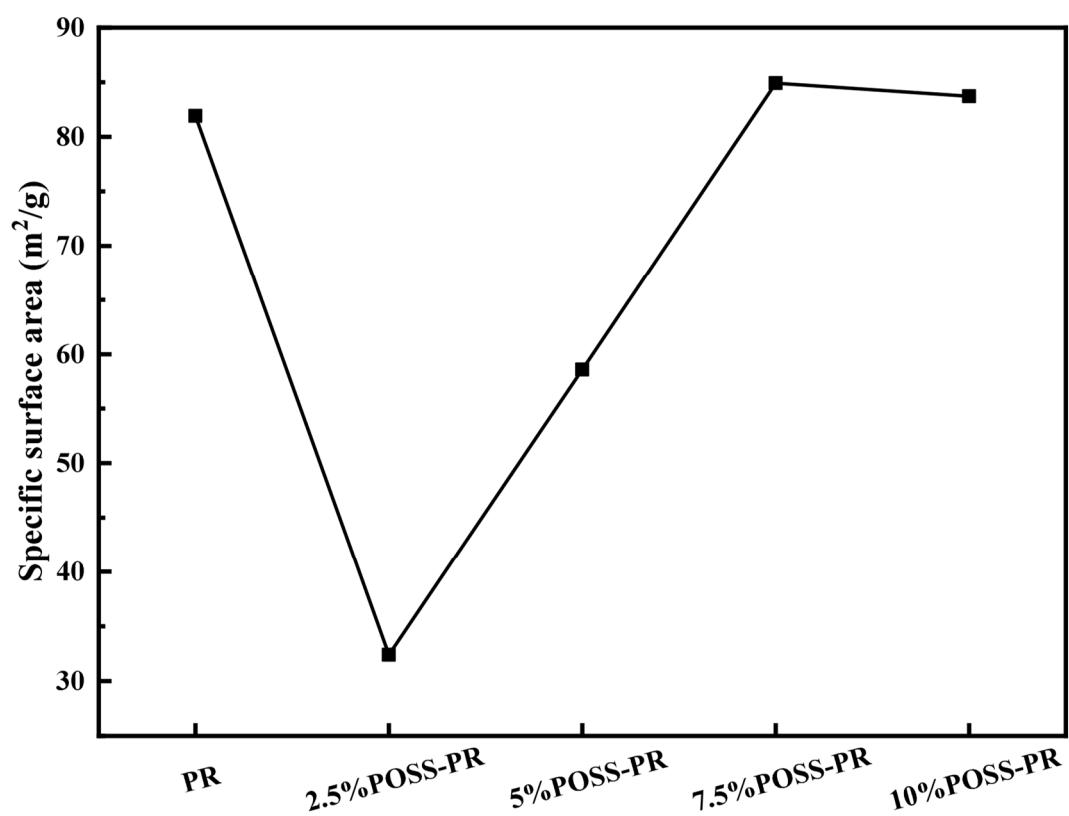


Figure. S6 Changes in specific surface area of phenolic resins with different POSS contents

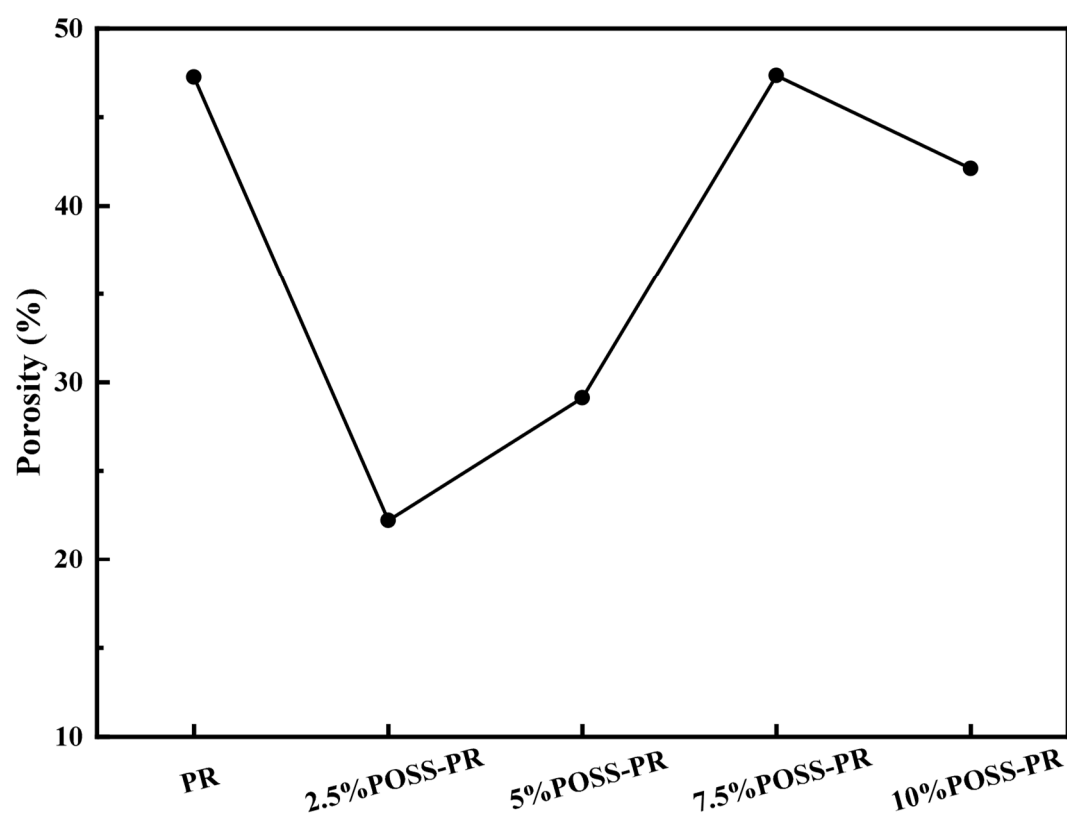


Figure. S7 Porosity changes of phenolic resins with different POSS contents

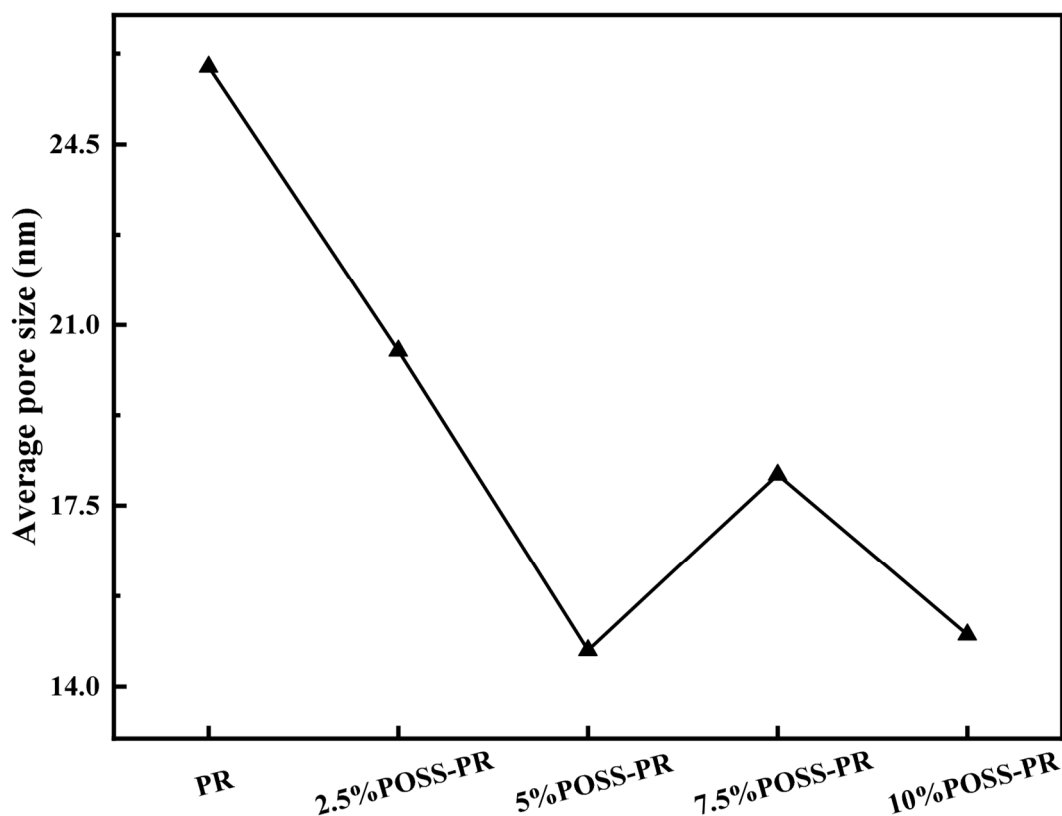


Figure. S8 Changes in average pore size of phenolic resins with different POSS contents

The N₂ adsorption-desorption curves of PR and POSS-PR are shown in Figure. S5. As the POSS content increases, the average pore size gradually decreases (Figure. S8). This is due to the POSS molecules occupying the voids within the phenolic porous network. However, the porosity and specific surface area show a pattern of first decreasing and then increasing (Figure. S6 and S7). This is because as the POSS content increases, the rigid structure of the POSS molecules exerts its support and expansion effect on the phenolic porous network, causing the hybrid network to expand further. The porosity-increasing effect caused by this expansion outweighs the porosity-reducing effect caused by the POSS molecules occupying the pores. Taken together, the higher porosity, specific surface area, and smaller pore size confirm the formation of the phenolic aerogel structure and indicate that the introduction of POSS molecules helps reduce the pore size of the phenolic aerogel material and increase the porosity and specific surface area.