Supplementary Information

Ultralight, Highly Thermal Insulating and Fire Resistant Aerogel by

Encapsulating Cellulose Nanofiber with Two-dimensional MoS₂

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Supplementary Figure 1 (a) Digital image of CNF solution with concentration of 1.0 wt.% after 30 days of preparation. (b) Raman spectra of the metallic phase MoS_2 . (c) Contact angle (Θ) of the metallic phase MoS_2 , is measured to be 22°. (d) Digital photograph of metallic phase MoS_2 solution 30 days after synthesis.



Supplementary Figure 2 (a, b) TEM images of CNF wrapped with metallic phase MoS_2 at different magnification.



Supplementary Figure 3 TGA curve of CNF/MoS₂ aerogel after burning.

Aerogel composition	CNF	CNF/MoS ₂	
Density (g/cm ³)	0.0045	0.0047	
Porosity (%)	99.69	97.36	
Surface area (m ² /g)	20.62	10.60	
Total pore volume (cm ³ /g)	0.038	0.026	

Supplementary Table 1 Microstructural properties of the CNF and CNF/MoS₂ aerogel

Sample	Heating flux (kW/m²)	Initial mass (g)	Final mass (g)	pkHRR (kW/m²)	THR (MJ/m²)
CNF/MoS ₂ aerogel	25	0.39	0.32	31	0.4
CMC paper ³⁷	25	1.14	/	94.4	1.53

Supplementary Table 2 Results of cone calorimetry test for CNF/MoS₂ aerogel



Supplementary Video 1: Snapshot from the X-ray microcomputed topography (μ CT) video of the CNF/MoS₂ aerogel reconstruction.



Supplementary Video 2: Ssnapshot from the X-ray microcomputed topography (μ CT) video of the CNF aerogel reconstruction.



Supplementary Video 3: Snapshot from video of the limiting oxygen index (LOI) test of the CNF/MoS_2 aerogel. This test is at an LOI of 34.7%, at which the nanocomposite aerogel slowly burned.



Supplementary Video 4: Snapshot from the video of the vertical burning test of the CNF/MoS_2 nanocomposite aerogel. The aerogel displayed self-extinguishing properties after the flame is removed.



Supplementary Video 5: Snapshot from the video of the vertical burning test of the CNF/MoS_2 nanocomposite aerogel using MoS_2 dipping method. The aerogel quickly burned after removal of the flame.



Supplementary Video 6: Snapshot from the video of the vertical burning test of the CNF aerogel. The aerogel quickly burned after removal of the flame.

Supplementary Materials 1: Thermal conductivity measurement

The bulk thermal conductivity of CNF/MoS₂ aerogel was measured using the ThermTest TPS 3500 thermal constants analyzer at 22 °C. Before thermal conductivity measurements, the CNF/MoS₂ aerogel was kept for 12 h at 55% relative humidity and 22 °C. The hot disk was sandwiched between two bulk CNF/MoS₂ aerogel samples (Supplementary Fig. 5a,b) and light pressure was applied to the sample sensor assembly to confirm interfacial contact for the test. Cylindrical samples 50 mm in diameter and 10 mm thick were used for the test. Measurements were made with 10 mW of output power for 10 seconds. The measurements were repeated four times for each sample.



Supplementary Figure 3 (a,b) Digital images of the measurement setup with the sensor sandwiched between the two cylindrical samples.