

## **Electronic Supplementary Information**

### **A 3D net-like structured fluorescent aerogel based on carboxy-methylated cellulose nanofibrils and carbon dots for a highly effective adsorbent and sensitive optical sensor of Cr(VI)**

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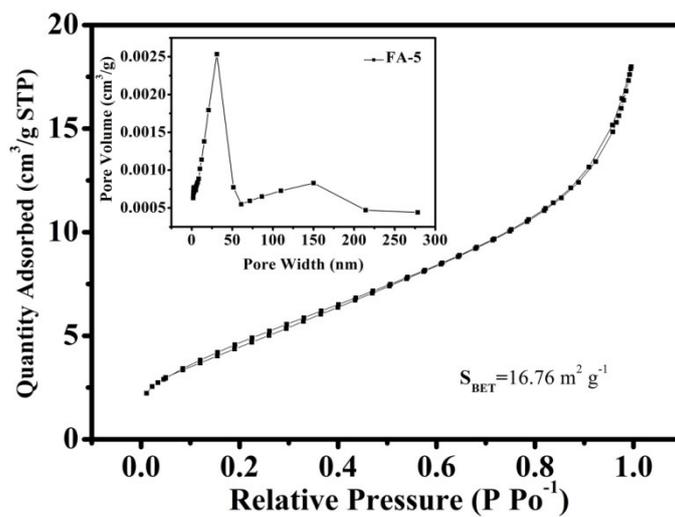
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Fig. S1-S3

Fig. S1 N<sub>2</sub> adsorption-desorption isotherm and the pore size distribution curve (inset) for the synthesized fluorescent aerogel.



The specific surface area and dominant pore size of the synthesized fluorescent aerogel were  $16.76 \text{ m}^2 \text{ g}^{-1}$  and 31 nm, respectively.

Fig. S2 FTIR spectra of the synthesized fluorescent aerogel before and after adsorption of Cr(VI).

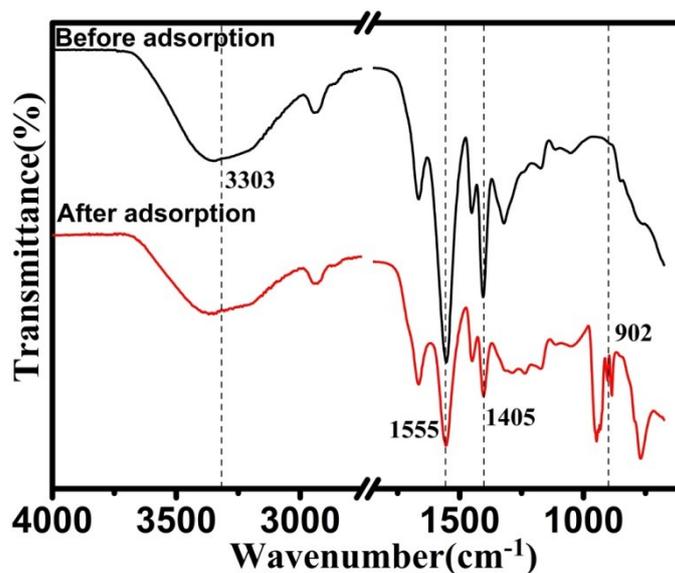
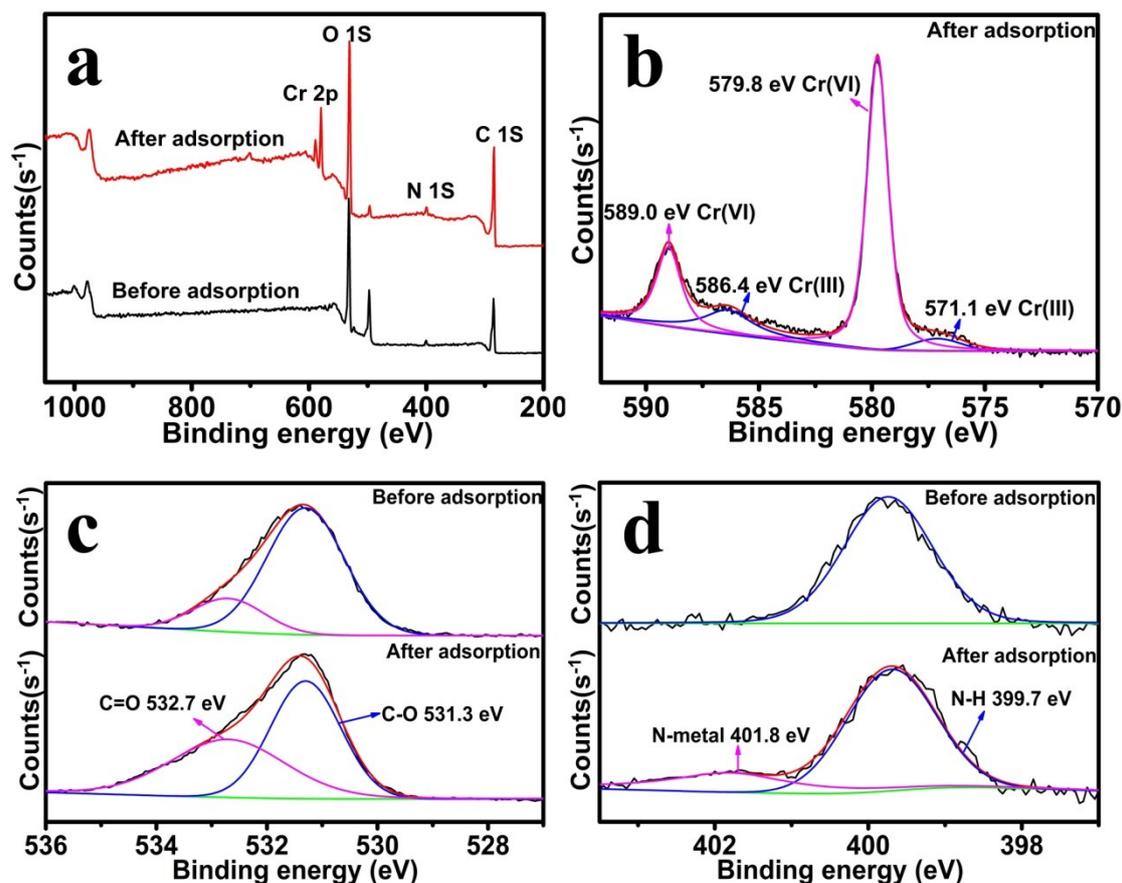


Fig. 2S shows FTIR spectra of the synthesized fluorescent aerogel before and after the adsorption of Cr(VI). After the adsorption of Cr(VI), the broad and strong band centered at  $\sim 3303\text{ cm}^{-1}$  (the superposition of the stretching vibrations of O–H and N–H groups)<sup>1</sup> decreased, two absorption bands at  $1555\text{ cm}^{-1}$  ( the bending vibration of N–H) and  $1405\text{ cm}^{-1}$  (the symmetrical stretching vibration of  $\text{COO}^-$ )<sup>2</sup> were weakened, while a new band at  $902\text{ cm}^{-1}$  (Cr species)<sup>3, 4</sup> appeared. These results indicated that amino, carboxyl, and hydroxyl groups of the synthesized fluorescent aerogel played a role in the adsorption process.

Fig. S3 XPS spectra of the synthesized fluorescent aerogel before and after adsorption of Cr(VI).



In order to further understand the adsorption mechanism, XPS analysis was also applied to characterize the changes of functional groups before and after adsorption. In the total XPS survey scans (Fig. 3S(a)), in addition to the characteristic peaks of C1s (284.6 eV), N1s (399.7 eV) and O1s (531.4 eV) for the synthesized fluorescent aerogel, the peaks of Cr2p (579.8 and 589.0 eV) were obviously observed after the adsorption of Cr(VI). Fig. 3S(b), Fig. 3S(c) and Fig. 3S(d) showed the high-resolution XPS results. As shown in Fig. 3S(b), the peaks at binding energy 579.8 and 589.0 eV were ascribed to Cr(VI), and the peaks at binding energy 571.1 and 586.4 eV were assigned to the Cr(III).<sup>5</sup> These facts confirmed the presence of Cr(VI) on the synthesized fluorescent aerogel and revealed the reduction of Cr(VI) to Cr(III) in some extent. Fig. 3S(c) illustrated that the integral area ratio of the two peaks of O1s at 532.7 eV (C=O) and 531.3 eV (C-O) for the synthesized fluorescent aerogel before adsorption and after adsorption increased from 0.242 to 0.862, indicating that the binding of Cr(III) occurred at O atoms of the carboxyl and hydroxyl groups.

Meanwhile, Fig. 3S(d) illustrated that before adsorption, there was only one typical N1s peak at 399.7 eV from the N atom in -NH<sub>2</sub> groups of the aerogel, while after the adsorption of Cr(VI), in addition to the peak of -NH<sub>2</sub> at around 399.7 eV, a new peak of N1s appears at 401.8 eV, which could be attributed to the formation of complexes between N and Cr(III).<sup>6</sup> These results indicated that Cr(VI) adsorption mainly began through electrostatic interaction, and then Cr(VI) partly reduced into Cr(III) which was complexed by the amino, carboxyl, and hydroxyl groups.

#### References:

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