Supplementary material

Optimum Selective Separation of Cu(II) Using 3D Ordered Macroporous Chitosan Films with Different Pore Sizes

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Figure S1. Zeta potential of 500nm3DOM-IICF

Zeta potential data is shown in Figure S1. Under acidic conditions, the amino group on the chitosan is protonated so that the surface of the film is positively charged. As the pH value increases, the amino protonation phenomenon is alleviated. An isoelectric point occurs at pH = 7. Since Cu(II) ions form a precipitate under alkaline conditions, alkaline conditions are not considered. Zeta potential data results are consistent with the results of adsorption experiments with different pH.



Figure S2. Adsorption of actual wastewater samples

A sample of wastewater is obtained at the effluent of the copper industry industry chain. We also measured the heavy metal content in the sample, and the results showed that Cu(II) content accounted for the highest proportion. The adsorption data is shown in Figure S2. The data shows that 3DOM-IICF has a highly selective adsorption capacity for Cu(II), and the heavy metal adsorbed is mainly Cu(II). And 3DOM-NIICF has a certain adsorption capacity for all heavy metal ions.



Figure S3. (a) spectrum of C 1s from XPS analysis; (b) spectrum of N 1s from XPS analysis; (c) spectrum of Cu 2p before adsorption from XPS analysis; (d) spectrum of Cu 2p before adsorption from XPS analysis; (e) total spectrum from XPS analysis

Figure S3. shows the XPS analysis of the 3DOM-IICF adsorption process for Cu(II). The three binding energies of 284.77, 286.47 and 288.37 eV in Figure a represent C-C, C-O and C=N bonds, respectively. The peak of C 1s was found to be shifted before and after the adsorption, which indicated that the adsorbent and the metal Cu(II) reacted chemically to form a complex¹. In the N 1s spectrum of Figure S3. b, the peak of the nitrogen atom in $-NH_2/-NH$ (400 eV) also shifts. Combined with the change of C=N in Figure S3. a, it indicates that Cu(II)-NH₂ complex is formed on the surface of the material². A comprehensive analysis of the Figure S3. c, d and e, the shift of the binding energy of N 1s and Cu 2p indicates that after the adsorption of Cu(II), in addition to - COOH, the lone pair of electrons in the $-NH_2$ group are also provided in $-NH_2$ and Cu(II)³.

EDS (Energy Dispersive Spectromete) is a good means of determining whether Cu(II) is actually adsorbed onto a material. By scanning the surface of the material by EDS, the Cu(II) adsorbed on the surface of the material can be clearly observed.



Figure S4. SEM and EDS images before and after adsorption of Cu(II) Table S1. Comparison of elemental composition results

Weight %	O K	Si K	Cu K	Au L
Before	35.75	47.8	0	16.46
After	40.32	34.47	9.41	15.79

The EDS data showed that the Cu(II) content in the 3DOM-IICF before adsorption was 0 %, and the Cu(II) mass ratio increased to 9.41 % after adsorption (see Table S1.). Figures S4 a and b are SEM and EDS plots of 3DOM-IICF before adsorption, figures S4 c and d are SEM and EDS plots of 3DOM-IICF after adsorption. The green mark in the EDS diagram is Cu(II). Cu(II) is uniformly distributed on the film, indicating that Cu(II) is actually adsorbed.

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