forests

MDPI

1. Draw the Standard Working Curve of Zn (II)

Preparation of zinc ion standard solution: accurately weigh 1.2520 g of reference zinc oxide solid and add it to a 1 l volumetric flask. After adding 10 ml of concentrated sulfuric acid, fix the volume to the scale mark with distilled water and shake well. The concentration of this standard solution is 1 g/L, which shall be diluted during subsequent use.

Prepare xylenol orange solution: accurately weigh 0.1500 g of xylenol orange into a 100 ml volumetric flask, add distilled water to volume to the scale mark, and shake well.

Preparation of acetic acid sodium acetate buffer solution: accurately measure 36 ml of glacial acetic acid in a 100 ml volumetric flask to volume, and shake well. Weigh and dissolve 200 g of anhydrous sodium acetate solid in water, heat, stir and dissolve it, transfer it to a 1 l volumetric flask, add 26 ml of the above glacial acetic acid solution, cool it, fix the volume to the scale mark, and shake it well.

Preparation of zinc ion standard curve: take 10 ml of zinc ion standard solution into a 1 l volumetric flask (C = 10 mg/L), add distilled water to volume to the scale mark, and shake well. Take 2.5 ml, 5 ml, 7.5 ml, 10 ml, 12.5 ml, 15 ml, 17.5 ml, 20 ml, 22.5 ml and 25 ml zinc ion solutions from them into 10 50 ml volumetric flasks respectively, add 10 ml acetic acid sodium acetate buffer solution and 2.5 ml xylenol orange solution successively, add water to the scale mark to volume, and shake well. Place it for 10 min, use a 1 cm cuvette at 570 nm to measure the absorbance with water as the reference, draw the standard curve with the concentration C (mg/L) of Zn (II) as the abscissa and the absorbance (ABS) as the ordinate, and prepare the Zn (II) standard curve equation, as shown in Figure S1.

2. Drawing of Mn (II) Standard Working Curve

Preparation of manganese ion standard solution: accurately weigh 3.6386 g of manganese chloride solid into a 1L volumetric flask, add distilled water to volume to the scale mark, and shake well. The concentration of this standard solution is 1 g/L, which shall be diluted during subsequent use.

Prepare potassium periodate solution: transfer 10ml concentrated nitric acid to a 100ml volumetric flask, add distilled water to volume to the scale mark, and shake well. Accurately weigh 2.0000 g of potassium periodate solid and dissolve it in 100 ml of the above nitric acid solution.

Prepare potassium pyrophosphate sodium acetate buffer solution: accurately weigh 230g of potassium pyrophosphate solid and 82G of anhydrous sodium acetate solid, dissolve them in water, heat, stir and dissolve them, transfer them to a 1L volumetric flask, cool them, fix the volume to the scale line, and shake them well.

Preparation of manganese ion standard curve: take 10 ml of manganese ion standard solution into a 1 l volumetric flask (C = 10 mg/L), add distilled water to volume to the scale mark, and shake well. Take 2.5 ml, 5 ml, 7.5 ml, 10 ml, 12.5 ml, 15 ml, 17.5 ml, 20 ml, 22.5 ml and 25 ml of manganese chloride solution from it into 10 50 ml volumetric flasks respectively, add 10 ml of potassium pyrophosphate sodium acetate buffer solution and 3 ml of potassium periodate solution in turn, add distilled water to the scale line for constant volume, and shake well. Place it for 20 minutes, use a 1 cm cuvette at 525 nm to measure the absorbance with water as the reference, draw the standard curve with the concentration C (mg/L) of Mn (II) as the abscissa and the absorbance (ABS) as the ordinate, and prepare the Mn (II) standard curve equation, as shown in Figure S1.

3. Drawing of Cu (II) Standard Working Curve

Preparation of copper ion standard solution: accurately weigh 3.9013 g of anhydrous copper sulfate solid into a 1L volumetric flask, add distilled water to volume to the scale mark, and shake well. The concentration of this standard solution is 1 g/L, and then accurately measure 1ml of 1g/L solution, transfer it to a 100ml volumetric flask, fix the volume, and the concentration of this solution is 10mg/L.

Weigh 1g of dicyclohexanone oxalyl dihydrazone solid sample into a 200ml beaker, add 100ml ethanol and heat it to 60oC. After dissolving the solid sample, transfer it to a 1000ml volumetric flask, and fix the volume with distilled water to the scale to obtain dicyclohexanone oxalyl dihydrazone solution with a mass concentration of 0.1%;

Prepare citric acid aqueous solution with a concentration of 0.5 g/ml, weigh 50g citric acid, put the solution into an 80ml beaker, heat and dissolve it, clean the beaker for many times, transfer the solution to a 100ml volumetric flask and fix the volume;

Mix ammonia and water according to the volume ratio of 1:1 to prepare ammonia solution, weigh 50ml ammonia and 50ml distilled water, mix evenly and set aside;

Accurately add 2.5ml, 5ml, 7.5ml, 10ml, 12.5ml, 15ml, 17.5ml, 20ml, 22.5ml and 25ml of Cu (II) (10 mg/L), 2ml of citric acid aqueous solution, 4ml of ammonia solution and 10ml of dicyclohexanone oxalyl dihydrazone solution (BCO) into 50ml volumetric flask, shake well and dilute to the scale. After 10min (the instrument is preheated for 10min), select 1ml cuvette, use the blank sample at 610nm as the reference solution, and measure its absorbance with an ultraviolet spectrophotometer. Carry out regression fitting on the obtained data and draw the standard working curve, as shown in Figure S1.

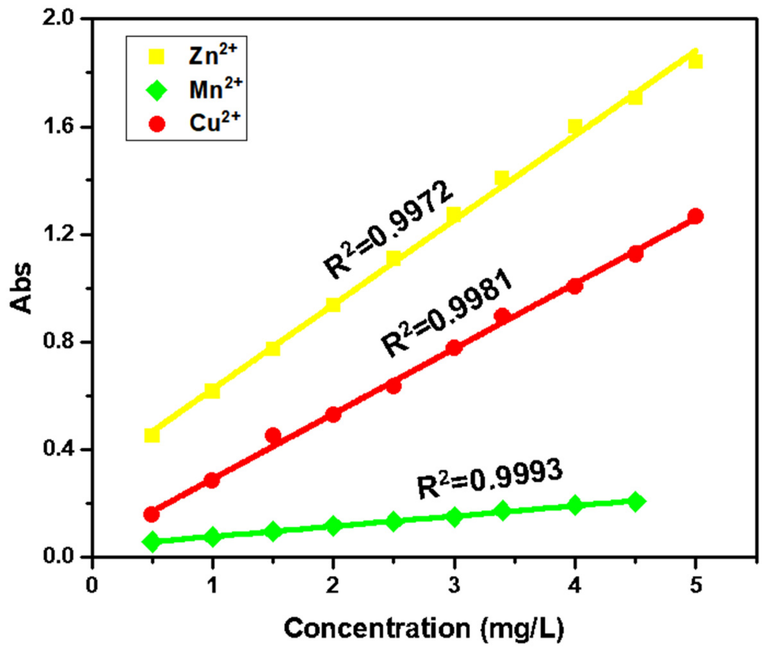

Figure S1. Drawing of heavy metal standard curve.

Table S1 shows the adsorption capacity of aerogel spheres for heavy metals,It can be seen from the Table S1, the adsorption capacity of aerogel spheres for Zn(II), Mn(II) and Cu(II) shows from individual to individual, among which the adsorption capacity of SC-8.5-TOCNF-1.5 for Zn(II), Mn(II) and Cu(II) has basically reached the maximum, there are 142.33 mg/g, 264.83 mg/g, 267.64 mg/g, respectively. Firstly, the increase of TOCNF content leads to the increase of active sites, thus increasing the adsorption capacity. However, when the adsorption capacity reaches the maximum, the amount of TOCNF continues to increase, the heavy metal adsorption capacity of samples began to decrease, There were two reasons for this: On the one hand, the agglomeration of part of TOCNF leads to the decrease of actual active adsorption sites; on the other hand, the increase of TOCNF content leads to the decrease of SP-MIC-C content, the decrease of structural stability of aerogel spheres, and the inward collapse of samples. The above shows that SC-8.5-TOCNF-1.5 has the best adsorption effect on Zn(II), Mn(II) and Cu(II).

Table S1. Drawing of heavy metal standard curve (adsorbent quality: 0.0500 g; adsorption time: 120 min; adsorption Concentration: 500 mg/g; adsorption Temperature: 60 oC; adsorption pH: 6.5).

Sample	Zn(II) (mg/g)	Mn(II) (mg/g)	Cu(II) (mg/g)
SC-1-TOCNF-0	96.65	78.63	52.75
SC-9.5-TOCNF-0.5	120.36	265.80	223.06
SC-9-TOCNF-1	131.61	267.32	241.63
SC-8.5-TOCNF-1.5	142.33	264.83	267.64
SC-8-TOCNF-2	140.49	246.03	260.79
SC-7.5-TOCNF-2.5	139.87	164.75	231.27