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Preparation of hydrophilic carbon-functionalized magnetic microspheres coated with chitosan and application in solid-phase extraction of bisphenol A in aqueous samples

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ABSTRACT

Hydrophilic carbon-functionalized magnetic microspheres coated with chitosan (Fe_3O_4@C@CHI) were synthesized for the first time. The hydrophilic carbonaceous layer on the Fe_3O_4 microspheres was formed by the hydrothermal reaction of Fe_3O_4 microspheres and glucose, and then chitosan was coated on surface of the carbon-functionalized microspheres by an easy zero-length reaction with 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide (EDC) and N-hydroxysuccinimide (NHS) as cross-linkers. The Fe_3O_4@C@CHI microspheres were spherical core–shell structure with uniform size at about 400 nm. The microspheres had a high saturation magnetization of 60.22 emu g^{-1}, which was sufficient for magnetic separation. The Fe_3O_4@C@CHI microspheres presented good hydrophilic property due to the abundant carboxyl and hydroxyl groups on the carbonaceous layer of the microspheres. The successful modification of chitosan on the surface of carbon-functionalized magnetic microspheres was validated by the adsorption of Eriochrome Black T (EBT). The prepared microspheres were applied as magnetic solid-phase extraction materials for extraction of bisphenol A (BPA) in aqueous samples. The extraction recoveries of spiked samples were in the range of 99.4–102.6% with low relative standard deviations of 2.8–3.9%. The results demonstrated that the novel Fe_3O_4@C@CHI microspheres were good candidate as magnetic SPE materials for extraction of analytes from aqueous samples.

1. Introduction

Recently, core–shell magnetic composites have attracted considerable attention because of their unique physical properties, nano-to-submicron size and outstanding surface chemistry properties of modified shell [1]. As the different functionally core–shell magnetic composites were achieved [2,3], they have shown more potential applications in catalyst [4], drug delivery [5], adsorption or separation science [6–8] and protein immobilization [9,10]. In addition, an increasing number of researches [11,12] were concentrated on the separation and enrichment of peptides or proteins with modified core–shell magnetic composites coupled with matrix assisted laser desorption ionization-time of flight MS (MALDI-TOF MS) as a trace detection technique due to their superparamagnetic property, large surface area and varies of surface modification. However, various features of different kinds of analytes require diverse and newly surface modification [13], and it remains a challenge for the long term development of modification techniques of magnetic materials in the future.

Chitosan is prepared from chitin, which is the second most abundant biopolymer next to cellulose, by partially deacetating its acetamido groups with a strong alkaline solution. It has been reported that chitosan's performances are good as an excellent biosorbent for metal ion, dyes and environmental pollutants [14,15]. Although chitosan which has a large number of amine groups is biocompatible, non-toxic, and easily obtained, it can only be dissolved in acid solution with low pH, which bring out great limitations to its practical applications. Several studies [16–20] have focused on the synthesis and application of modified Fe_3O_4 nanoparticles. More recently, Zhang et al. [21] prepared hydrophilic carbon coated Fe_3O_4 nanoparticles (Fe_3O_4@Carbon, Fe_3O_4@C) as a sorbent. Their research mentioned that there were great quantity of carboxyl groups and hydroxyl groups on the surface of Fe_3O_4@C nanoparticles, which were used in the solid-phase extraction of polycyclic aromatic hydrocarbons in the article. Qi et al. [22] also reported the synthesis of Fe_3O_4@C@SnO_2 core–shell microspheres, which make good use of coated Fe_3O_4 with thin hydrophilic carbonaceous layer possessing numerous hydrophilic groups such as —COOH and —OH. The surface of the Fe_3O_4@C microspheres was then modified with SnO_2 nanoparticles, which was

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applied to phosphoproteomics. Given the advantage of chitosan and Fe_3O_4@C mentioned above, it could be possible to connect the amino group of the chitosan with the carboxyl group on the surface of Fe_3O_4@C microspheres by an easy cross-linker reaction with 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide (EDC) and N-hydroxysuccinimide (NHS) which is well known in bioconjugation.

To the best of our knowledge, although some studies on the synthesis, characterization and application of Fe_3O_4@C and Fe_3O_4@Chitosan have been reported, chitosan coated carbon-functionalized magnetite microspheres as Fe_3O_4@C@CHI were prepared for the first time by a non-toxic environmental friendly method, which had good hydrophilic property even in neutral pH, strong superparamagnetic property and excellent adsorption property. The hydrophilic carbonaceous layer on the Fe_3O_4 microspheres was formed by the hydrothermal reaction of Fe_3O_4 microspheres and glucose. Chitosan was coated on the carbon-functionalized surface of microspheres by an easy zero-length reaction with 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide (EDC) and N-hydroxysuccinimide (NHS) as cross-linkers. The prepared microspheres had strong superparamagnetic property from Fe_3O_4 core, which contributed to the convenient isolation of extracted analysts from matrix solution in SPE procedure. The developed magnetic SPE method was more rapid and portable, and lower cost for operation. These microspheres were used for adsorption of Eriochrome Black T (EBT) to validate the successful modification of chitosan on the surface of carbon-functionalized magnetic microspheres, and applied to the extraction of BPA in aqueous samples.

2. Experimental

2.1. Materials

All reagents were of analytical grade and used without further purification. $FeCl_3{\cdot}6H_2O$, NaAc, ethanol, ethylene glycol were obtained from Beijing Chemical Reagent Company (Beijing, China). Glucose, bisphenol A (BPA) and chitosan were obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Poly(ethylene glycol) average M.W. 1500 (PEG 1500) was purchased from Merck Company (Darmstadt, Germany). 1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide·HCl (EDC·HCl) and N-hydroxysuccinimide (NHS) were obtained from GL Biochem Ltd. (Shanghai, China). Eriochrome Black T (EBT) was purchased from Yongjia Fine Chemical Industry Factory (Zhejiang, China). All water was freshly deionized using an ultraviolet ultrapure water system (18.3 MΩ cm, Barnstead, IO, USA).

2.2. Apparatus

Powder X-ray diffraction (XRD) patterns were obtained on a D8 Advance X-ray diffractometer (Bruker, Germany). Fourier transform infrared (FT-IR) measurement was carried out with a PerkinElmer 100 FT-IR spectrometer (MA, USA). Transmission electron microscopy (TEM) images were recorded by a H-7650B electron microscope operating at 80 kV (Hitachi, Japan). Scanning electron microscope (SEM) images were determined on a FEI Quanta 200 Microscope (FEI, Czech) by operating at 20 kV. Magnetization curves were measured using a 7307 Vibrating Sample Magnetometer (LakeShore, America). X-ray photoelectron spectroscopy (XPS) data were detected with a PHI-5300 ESCA X-ray photoelectron spectroscopy (PHI, America). The UV spectrum was acquired by a UV–vis U-3900 spectrophotometer (Hitachi, Japan). The chromatographic analysis was performed with a HPLC system (Hitachi, Japan).

2.3. Synthesis of chitosan modified Fe_3O_4@C microspheres

2.3.1. Synthesis of Fe_3O_4 microspheres

The Fe_3O_4 microspheres were synthesized using a solvothermal reaction described as follows [23]: $FeCl_3{\cdot}6H_2O$ (1.35 g, 5 mmol) was totally dissolved in ethylene glycol (40 mL), followed by the addition of NaAc (3.6 g) and polyethylene glycol (PEG 1500, 1.0 g). The mixture was stirred vigorously to obtain a clear yellow solution and then the solution was sonicated for 5 min for bubble removal. The resulting solution was transferred into a 50 mL Teflon-lined stainless-steel autoclave. The sealed autoclave was heated and maintained at 200 °C for 8 h, and allowed to cool to room temperature. The black products of Fe_3O_4 microspheres were collected with an external magnet field, followed by washing several times with deionized water and ethanol. Then the Fe_3O_4 microspheres were dried at 60 °C in an oven under vacuum for 6 h.

2.3.2. Synthesis of Fe_3O_4@C core/shell microspheres

As shown in Scheme 1, the Fe_3O_4@C microspheres were synthesized by a simple hydrothermal reaction [22]: the synthesized Fe_3O_4 microspheres (0.2 g) were sonicated in 40 mL of 0.1 M HNO_3 solution for 10 min, followed by washing with deionized water for three times. Then the treated Fe_3O_4 microspheres were redispersed in 35 mL of 3.6 g glucose aqueous solution and sonicated for 10 min. The suspension was transferred into autoclaves and kept at 180 °C for 6 h, then cooled to room temperature. The resulting Fe_3O_4@C microspheres were isolated with an external magnetic field and washed with deionized water and ethanol for six times, then dried at 60 °C for 6 h.

2.3.3. Synthesis of Fe_3O_4@C@CHI microspheres

The Fe_3O_4@C@CHI microspheres were prepared by an easy zero-length cross-linker reaction with EDC·HCl and NHS (Scheme 1) [24]. 0.3 g of the prepared Fe_3O_4@C microspheres was dispersed in deionized water. The surface of Fe_3O_4@C microspheres was preactivated by adding EDC·HCl PB buffer and NHS PB buffer for incubation. Chitosan was firstly dissolved in 0.5% acetic acid, then was diluted with PB and deionized water. The final chitosan solution was added into the preactivated Fe_3O_4@C microspheres solution, the mixture was shaken for 2 h. Afterwards, the obtained Fe_3O_4@C@CHI microspheres were isolated with an external magnet field, then washed with deionized water and dried at 60 °C in an oven prior to use.

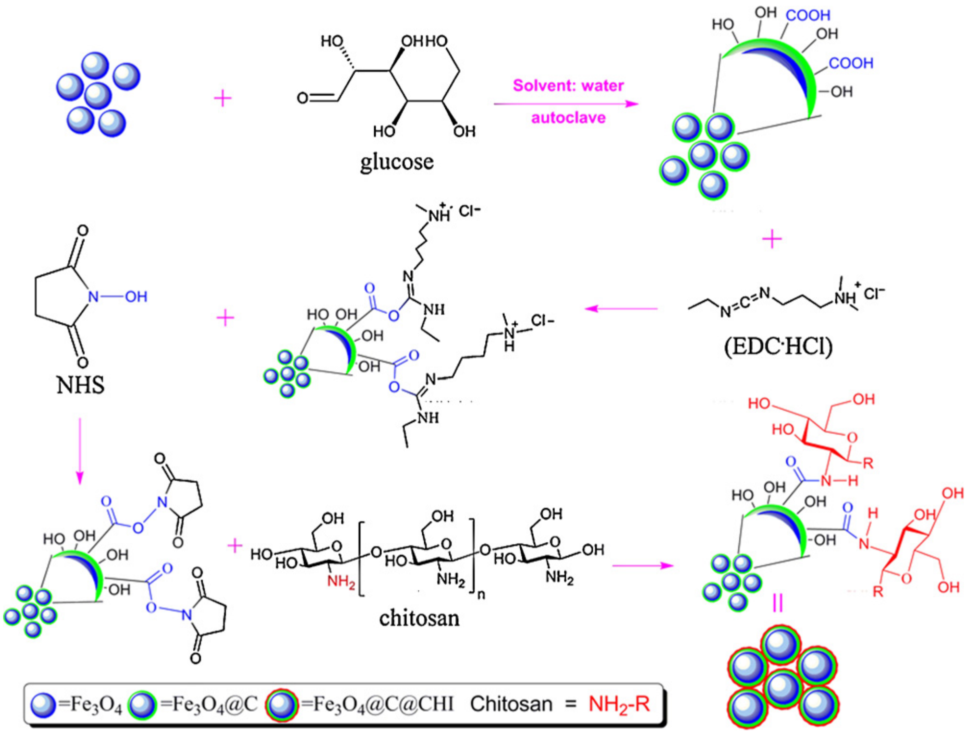
2.4. Evaluation of Fe_3O_4@C@CHI microspheres

EBT was dissolved in distilled water with the concentration of $50\,mg\,L^{-1}$. Then 20 mg of the synthesized Fe_3O_4@C@CHI microspheres was added into the EBT solution. The resulting mixture was sonicated at room temperature for less than 1 min to form a homogeneous dispersion solution. The microspheres were separated rapidly from the solution under an external magnetic field after 2 min. The clear EBT solution was measured by a UV–vis U-3900 spectrophotometer.

2.5. Determination procedure of BPA

2.5.1. SPE procedures for BPA

The SPE procedure consisted of three steps. BPA was dissolved in distilled water at concentration of $0.5\,mg\,L^{-1}$, and 20 mg of Fe_3O_4@C@CHI microspheres was added into the BPA solution. The mixture was sonicated at room temperature for less than 1 min to form a homogeneous dispersion solution. Then, the microspheres were isolated rapidly from the solution under an external magnetic field after 20 min. Finally, the BPA was eluted from the



Scheme 1. Synthesis steps of Fe_3O_4@C@CHI.

Fe_3O_4@C@CHI microspheres with 1 mL of methanol. 10 μL of the BPA eluate was analyzed by HPLC-FLD.

2.5.2. Chromatographic conditions

The HPLC-FLD system consisted of a fluorescence detector with a standard flow cell, a quaternary pump with online degasser, an autosampler and a column oven. The separation was performed on a reversed phase C_{18} column (SinoChrom ODS-BP, 5.0 μm, 4.6 mm × 250 mm, Elite, Dalian, China). The mobile phase was a mixture of acetonitrile–pure water (60:40, v/v). The separation procedure was finished within 15 min at flow rate of 1 mL min^{-1}. The column oven was controlled at 30 °C, and the detection wavelengths were selected at 230 nm and 301 nm for excitation and emission, respectively. Samples were filtered through 0.45 μm membrane prior to injection.

3. Results and discussion

3.1. The characterization of magnetic particles

The TEM and SEM images of the synthesized Fe_3O_4, Fe_3O_4@C and Fe_3O_4@C@CHI microspheres are shown in Fig. 1. As shown in Fig. 1A of the TEM image of Fe_3O_4@C@CHI microspheres, a gray shell surrounding the dark core of Fe_3O_4 is observed which demonstrates the uniform coating of carbon and chitosan on the surface of the Fe_3O_4@C@CHI microspheres with ca. 20 nm thickness. Fig. 1B is the SEM image of the synthesized Fe_3O_4 particles, which are spherical shape and have uniform diameter of about 380 nm. Fig. 1C and D are the synthesized Fe_3O_4@C and Fe_3O_4@C@CHI microspheres, respectively. The microspheres after carbon function and chitosan coating remained good granularity and dispersivity.

FT-IR evidence for successful synthesis of Fe_3O_4@C@CHI microspheres was demonstrated in Fig. 2. Typical peaks assigned to the Fe—O stretching were visible at around 582 cm^{-1} for Fe_3O_4, Fe_3O_4@C, and Fe_3O_4@C@CHI. From Fe_3O_4@C spectrum, the bands at 1617 cm^{-1} and 1706 cm^{-1} may belong to the C═C and C═O from —COOH. The peak of 3500 cm^{-1} from —OH in Fe_3O_4@C@CHI spectrum which was a little stronger than in Fe_3O_4 and Fe_3O_4@C spectrum may result from the C—OH of chitosan. Also, 1011 cm^{-1} indicates the existence of C—O from C—OH, which may be from the C—OH of chitosan. Meanwhile, C—O stretching from C—O—C of chitosan was obtained at around 1165 cm^{-1} for Fe_3O_4@C@CHI microspheres. The XPS detection results were listed in Table 1. The coexistence of C, N, O and Fe elements on the surface of the Fe_3O_4@C@CHI microspheres indicated the successful synthesis of Fe_3O_4@C@CHI microspheres.

The XRD of the Fe_3O_4, Fe_3O_4@C and Fe_3O_4@C@CHI microspheres (Fig. 3), compared with the standard card of Fe_3O_4, shows that the microspheres prior to modification were Fe_3O_4 crystal while after modification, the microspheres remained the Fe_3O_4 crystal and the outer shell was amorphous. Fig. 4 shows the magnetization curve of Fe_3O_4, Fe_3O_4@C and Fe_3O_4@C@CHI microspheres. Because of the coating of carbon and chitosan, the maximum magnetization has a little decrease. The Fe_3O_4@C@CHI microspheres have highly saturation magnetization of 60.22 emu g^{-1} compared with small size of magnetic materials [25]. No hysteresis, remanence and coercivity, displayed in Fig. 4, illustrated the superparamagnetic characteristic of the magnetic composites.

3.2. Evaluation of Fe_3O_4@C@CHI microspheres

Fig. 5B shows that the microspheres can be easily and fully mixed in the EBT solution because of the hydrophilic characterization of the interior carbon shell. Chitosan on the surface of the

Table 1
X-ray photoelectron spectroscopy (XPS) results of Fe_3O_4@C@CHI.

Element	Molar ratio (%)
C	60.16
N	2.67
O	34.4
Fe	2.77

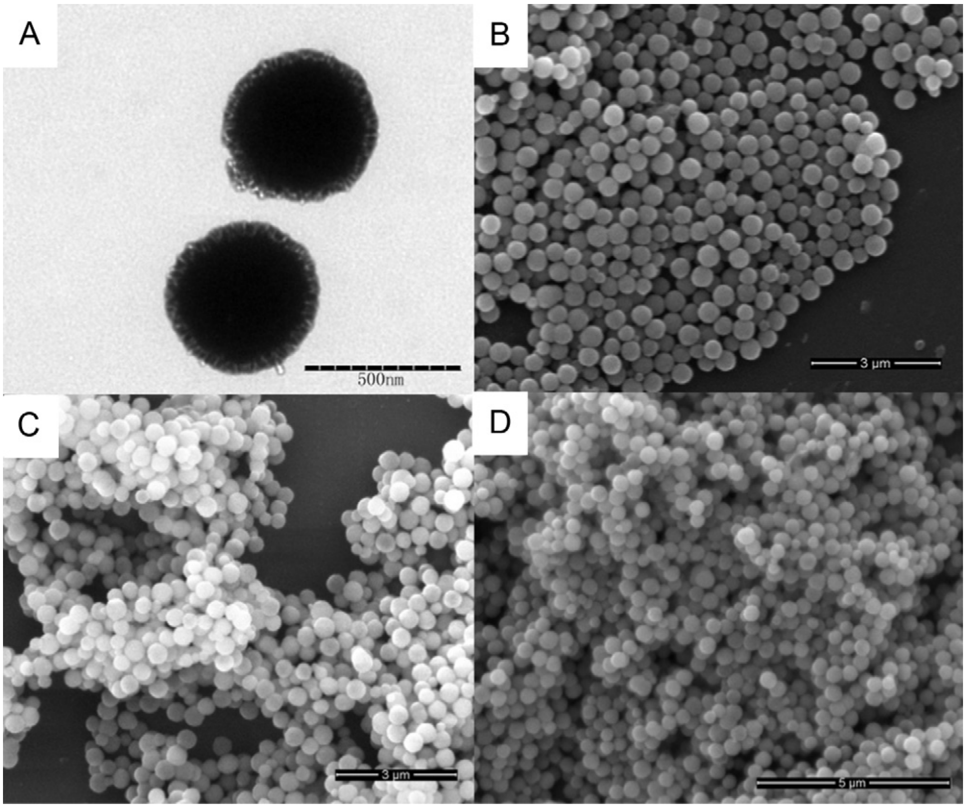


Fig. 1. TEM image of Fe_3O_4@C@CHI (A) and SEM images of Fe_3O_4 (B), Fe_3O_4@C (C), and Fe_3O_4@C@CHI (D).

shell can totally act its adsorption ability in the neutral pH (The pH value of EBT solution was measured to be 7.08.). Fig. 5A–C shows the process of using synthesized Fe_3O_4@C@CHI microspheres to absorb EBT in aqueous solution. The original EBT water solution had purple color (Fig. 5A). As shown in Fig. 5B, 20 mg Fe_3O_4@C@CHI microspheres was added into the 50 mg L^{-1} EBT solution, and a homogeneously dispersive solution was obtained. Under an external magnetic field, the Fe_3O_4@C@CHI microspheres with adsorbed EBT dye were isolated and so the purple color of original EBT solution disappeared (Fig. 5C). This phenomenon indicated that EBT was almost adsorbed by the microspheres. As a comparison, Fe_3O_4@C microspheres were added into the EBT solution. After isolation under an external magnetic field, the microspheres just made the solution color turn into scarlet shade (Fig. 5D). The different colors of the two different EBT solutions after being adsorbed by the Fe_3O_4@C@CHI microspheres and Fe_3O_4@C microspheres indicated that chitosan coating exhibited excellent adsorptive property.

Further, in order to investigate the adsorption capabilities and adsorption rate of the Fe_3O_4@C@CHI microspheres, different amounts of 5, 10, 20, 30 mg dried Fe_3O_4@C@CHI microspheres were added into 5.0 mL EBT at 50 mg L^{-1}concentration aqueous solution. Fig. 5E shows that less amount of Fe_3O_4@C@CHI microspheres would slow down the adsorption rate. 5.0 mg microspheres

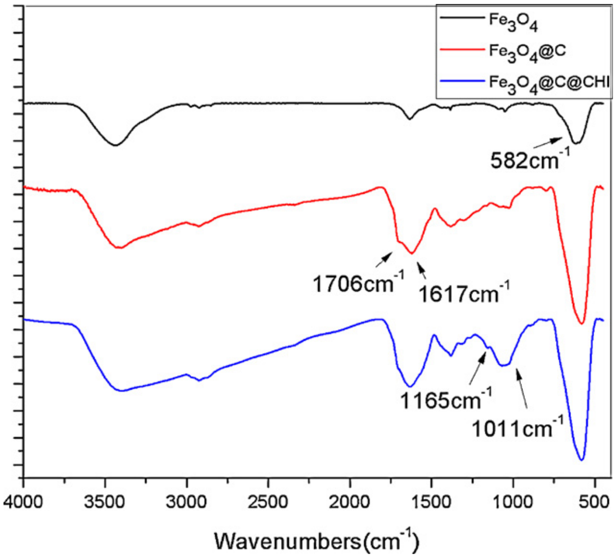


Fig. 2. FT-IR spectra of Fe_3O_4, Fe_3O_4@C, and Fe_3O_4@C@CHI microspheres.

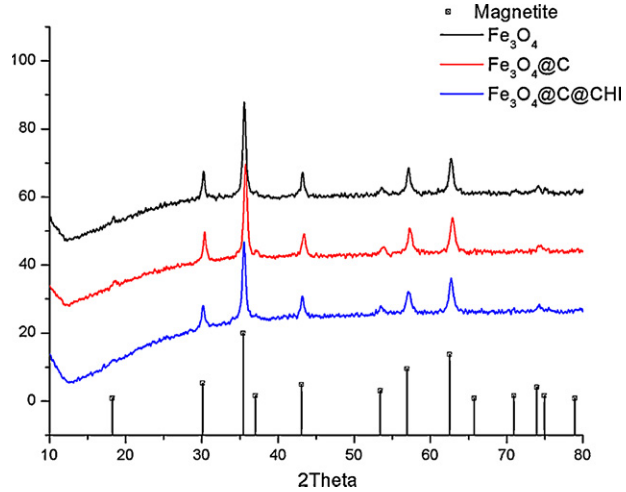


Fig. 3. XRD patterns of Fe_3O_4, Fe_3O_4@C, Fe_3O_4@C@CHI and the standard card of Fe_3O_4.

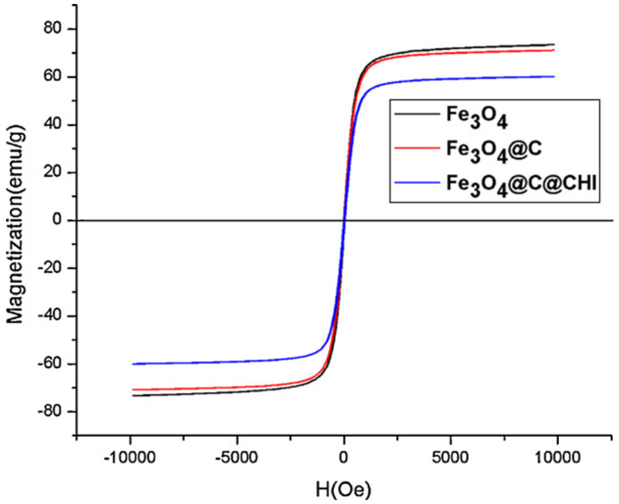


Fig. 4. Room-temperature magnetization curves of Fe_3O_4, Fe_3O_4@C and Fe_3O_4@C@CHI microspheres.

can only reach an adsorption capacity of 70%, while 20 or 30 mg Fe_3O_4@C@CHI microspheres can reach an adsorption capacity of 97% within 180 s. The rapid absorption saturation owed to the submicron size and large surface area of the microspheres.

3.3. *Effect of the adsorption time for BPA in aqueous samples*

It needs some time to adsorb analytes from the sorbent, so the investigation of the influence of the adsorption time (5, 8, 10, 20, 25, and 40 min) was carried out. The amount of the Fe_3O_4@C@CHI microspheres was 20 mg and it was added into 0.5 mg L^{-1} spiked BPA aqueous sample. The whole SPE procedure was carried out similar with experiment Section 2.5.1 but with different adsorption time. As shown in Fig. 6, 20 min is enough for sufficient absorption of BPA from the aqueous solution. Therefore, 20 min was chosen as the optimized adsorption time. Compared with the other extraction procedure for BPA (traditional SPE procedure, flash chromatography), the developed magnetic SPE method was more rapid and portable, and lower cost for operation [26,27].

3.4. *Effect of desorption solvent for BPA in aqueous samples*

Common organic solvents usually perform effective for desorption of organic analytes. The whole SPE procedure was carried out similar with experiment Section 2.5.1, and six different organic solvents (methanol, ethanol, acetonitrile, toluene, 1-hexanol, and cyclohexane) were used to elute BPA from Fe_3O_4@C@CHI microspheres as illustrated in Fig. 7. Methanol was suitable eluent for the SPE procedure.

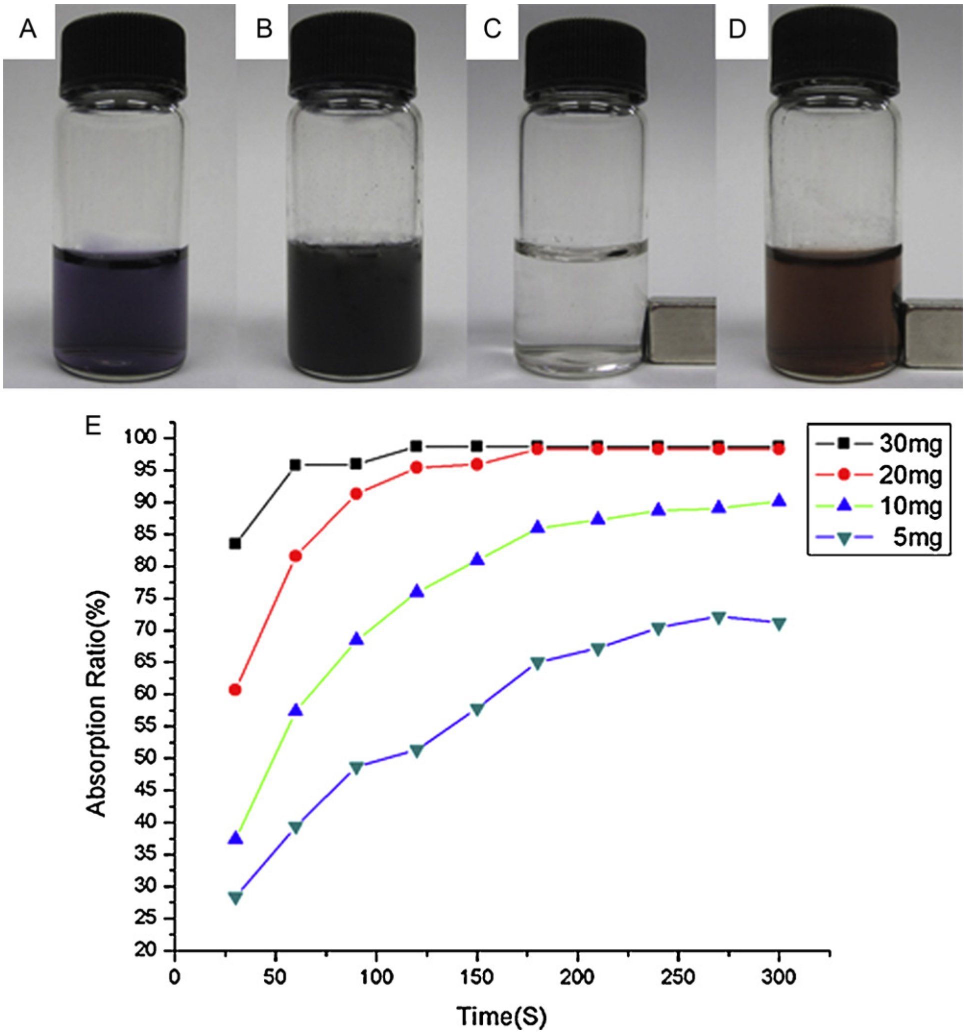


Fig. 5. Adsorption ratio of Eriochrome Black T with different amounts of Fe_3O_4@C@CHI microspheres and images of the adsorption process.

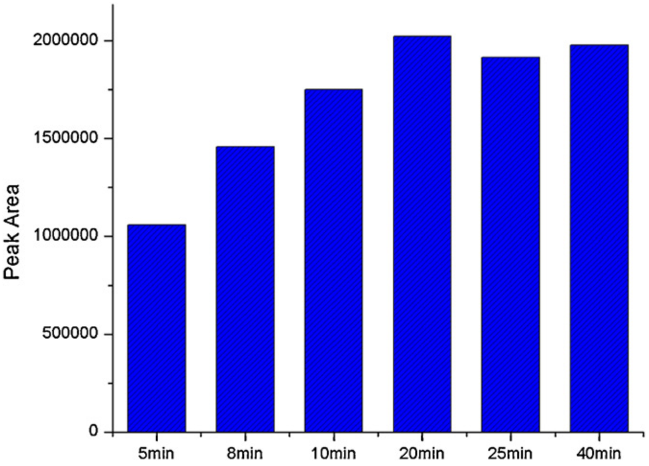


Fig. 6. The effect of the time of absorption on the extraction performance (adsorbent: 20 mg Fe_3O_4@C@CHI microspheres, sample: 1 mL 0.5 mg L^{-1} spiked BPA in aqueous sample and desorption solvent: 1 mL methanol).

3.5. Evaluation of the extraction performance for BPA in aqueous samples

Under the optimum condition, the linearity range, limit of detection, repeatability of the method were investigated. The calibration equation was $y = 3311336x - 165379$. Good correlation coefficient (R^2) with excellent linearity for target analytes was 0.997. And the linearity range of analysis of BPA was 5–5000 μg L^{-1}. Furthermore, the limit of detections (S/N = 3) was 1.92 μg L^{-1} and the repeatability was calculated by the three repetitive determinations of 500 μg L^{-1} standard BPA sample.

3.6. Determination of BPA in spiked tap water samples

Tap water was chosen to validate the extraction performance of the magnetic SPE method with Fe_3O_4@C@CHI microspheres. No BPA was detected in the original tap water. Tap water samples spiked with BPA at three concentration levels of 50, 100, 500 μg L^{-1} were detected, respectively. The recoveries and reproducibility of the method are listed in Table 2. The recoveries are in the range of 99.4–102.6% demonstrating good extraction efficiency of the developed magnetic SPE procedure.

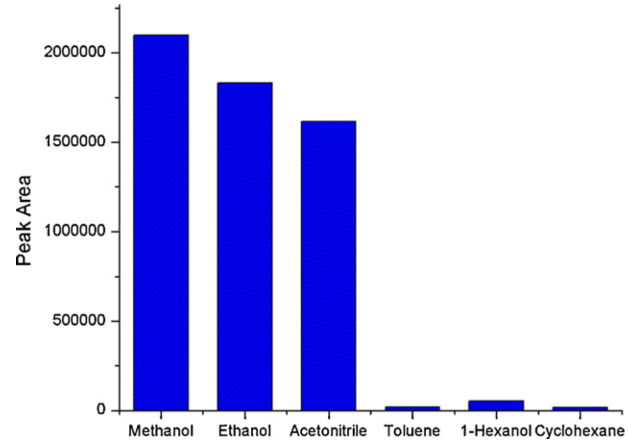


Fig. 7. The effect of desorption solvent on the extraction performance (adsorbent: 20 mg Fe_3O_4@C@CHI microspheres, sample: 1 mL 0.5 mg L^{-1} spiked BPA in aqueous sample and adsorption time: 20 min).

Table 2
Recoveries (%) and R.S.D. (%) of BPA with spiked tap water samples.

BPA spiked in water sample (μg L^{-1})	Recovery (%)	R.S.D. (%)
50	99.4	2.8
100	102.6	3.2
500	101.3	3.9

4. Conclusions

In the present study, one kind of hydrophilic carbon-functionalized magnetic microspheres coated with chitosan (Fe_3O_4@C@CHI) has been initially synthesized using an easy zero-length cross-linker reaction. Glucose was firstly applied to carbon-functionalized Fe_3O_4 microspheres for linking of chitosan. These Fe_3O_4@C@CHI microspheres, presenting good hydrophilic property even in neutral pH and large surface area, provided high extraction efficiency to analytes. These Fe_3O_4@C@CHI microspheres exhibited good extraction efficiency for EBT dye and BPA in aqueous solutions. The Fe_3O_4@C@CHI microspheres are good potential sorbent for development of magnetic SPE method.

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