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Samarium (III) adsorption on bentonite modified with *N*-(2-hydroxyethyl) ethylenediamine

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ABSTRACT

A new material has been synthesized using dry process to activate bentonite followed by *N*-(2-hydroxyethyl) ethylenediamine connecting chlorosilane coupling agent. The synthesized new material was characterized by elemental analysis, FT-IR and thermogravimetry which proved that bentonite was successfully modified. The most interesting trait of the new material was its selective adsorption for rare earth elements. A variety of conditions of the new material were investigated for adsorption. The optimal conditions were determined with respect to pH and shaking time. Samarium (Sm) was quantitatively adsorbed at pH 4 and shaking time of 2 min onto the new material. Under these conditions the maximum static adsorption capacity of Sm(III) was found to be 17.7 mg g^{-1}. The adsorbed Sm(III) ion were quantitatively eluted by 2.0 mL 0.1 mol L^{-1} HCl and 5% CS $(NH_2)_2$ solution. According to IUPAC definition, the detection limit (3σ) of this method was 0.60 ng mL^{-1}. The relative standard deviation (RSD) under optimum conditions was less than 3% ($n = 8$). The new material also was applied for the preconcentration of trace Sm(III) in environmental samples with satisfactory results.

1. Introduction

Since ancient times, bentonites have been used for a plethora of applications due to their large adsorption capacity and swelling characteristics in aqueous suspensions [1,2]. Also its cation exchange capacity and adsorptive affinity for organic and inorganic ions are considered two most promising candidates for use in decontamination and disposal of high-level heavy metal wastes [3,4]. Nowadays, with the development of science and technology, the improvement of research methods and means, in-depth understanding of bentonite composition, nature and performance, the increase of the demand for mineral raw materials with necessity of the social development especially, the application of bentonite becomes more and more extensive. Bentonites are used, for example, as clarifying agents in the food industries, as filler for drilling mud in oil wells or to alter rheological properties of coating materials, pharmaceuticals and soil [5]. In 1972, R.E. Grim put forward the broad concept of bentonite: bentonite is a montmorillonite mineral as the main component of the rock and it is the content of montmorillonite mineral reach to available clay or clay rock. The inner layer of bentonites is composed of an octahedral sheet situated between two SiO_4 tetrahedral sheets 2:1 type silicate minerals [6]. The silicon ion of silicon-oxy tetrahedron and the aluminum ion of alumina octahedral sheet were substituted by other unequal cations (Fe^{3+}, Fe^{2+}, Zn^{2+}, Mn^{2+}, Li^{+}, etc.), this phenomenon was called crystal lattice permutation, which leads to charge difference. The result of charge difference is that the chemical bond within wafer and crystal layer is more in favor of electrovalent bond, so that crystal layer possesses the capacity to adsorb cations.

The rare earth elements (REEs) are particularly useful trace elements. Today there is an increasing need for rare earth in various fields, the optical, the electronics, the ceramic, the nuclear and geochemistry [7]. However, the content of the REEs is incomplete (17 elements account for 0.0153% of total crust). So, it becomes more and more important and necessary to recycle and reuse REEs from industry and environment. Among the 17 rare earth elements, samarium was widely used as neutron absorber in nuclear industry, as a chemical reagent in organic synthesis in chemistry, and most important of all, in radio pharmacy (radioactive samarium-153 is used in medicine to treat the severe pain associated with cancers that have spread to the bones) [8,9].

A separation method of REEs from a solution containing many REEs has been developed [10]. Nowadays, solid-phase extraction (SPE) has become the most common method because of its advantages of high recovery, short extraction time, high enrichment factor, low cost and low consumption of organic solvents over liquid-liquid extraction [11,12].

In this paper, bentonite was treated with hydrochloric acid and modified by *N*-(2-hydroxyethyl) ethylenediamine the first time. The new material was characterized by FT-IR, elemental anal-

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ysis and thermogravimetric analysis (TGA). In SPE method, the new material showed great selectivity, relatively high adsorption capacity, less equilibrium time and easy elution for Sm(III). Several parameters that could affect the adsorption and elution efficiency of target ions were studied by batch and column modes. The new material was applied to adsorb Sm(III) from environmental samples.

2. Experiments

2.1. Chemicals and reagents

Analytical reagents and chemicals of spectral purity were used for all experiments and doubly distilled water was used in the whole process. Standard labware and glassware used were repeatedly cleaned with HNO_3 and rinsed with double distilled water, according to a published procedure [13]. Standard stock solutions of Sm(III) (1 mg mL^{-1}) were prepared as follows: spectral pure grade samarium oxide was dissolved into hydrochloric acid (The First Reagent Factory, Shanghai, China) with double distilled water to the target volume. Before using, Sm(III) (1 mg mL^{-1}) should be diluted to the various concentrations between 10 and 100 $\mu g\ mL^{-1}$. The pH of the solutions was adjusted to expected values with 0.1 M HCl or 0.1 M $NH_3{\cdot}H_2O$.

Bentonite (BT) (Tianjin Institute of Fine Chemicals retrocession) was ground, sieved through a 63 μm size sieve and samples collected from under the sieve and dried in an oven at 110 °C for 2 h before using [14]. *N*-(2-Hydroxyethyl) ethylenediamine (NHED) was provided by Alfa Company. 3-Chloropropyl trimethoxy silane (WD-30) was obtained from Hubei Wuhan University Silicone New Material Co., Ltd. (Wuhan, China).

2.2. Instruments and apparatus

All metal ions were determined by an IRIS advantage ER/S inductively coupled plasma spectrometer, Thermo Jarrel Ash (Franklin, MA, USA). The instrumental parameters were those recommended by the manufacturer. The operation conditions and the wavelengths were summarized in Table 1. The wavelength selected was Sm(III): 442.434 nm. A pHs-3c digital pH meter (Shanghai lei ci Device Works, Shanghai, China) was used to measure the pH value. Fourier Transform Infrared (FT-IR) spectra (4000–200 cm^{-1}) in KBr were recorded using Nicolet Nexus 670 FT-IR spectrometer, Nicolet (Madison, WI, USA). The flow rate of solution was controlled by an YL-110 peristaltic pump (General Research Institute for Nonferrous metals, Beijing, China). A reciprocating shaker batch SHZ-88-1 (Taicang Laboratorial Equipment Factory, Jiangsu, China) was used. A Vario EL element analyzer model Elementar Analysensysteme (Hanau, Germany) was used for elemental analysis. The thermogravimetry (TG) curves were obtained by using a STA449C (Netzsch, Germany). The measurements were carried out under flowing nitrogen atmosphere with a flow rate of 200 mL min^{-1} and the temperature ranged from 24 to 1000 °C in aluminum crucible. The heating rates were 20 °C min^{-1} and the sample masses were in the range of 6–10 mg. Highly-sintered Al_2O_3 was used as the reference material. A glass micro-column (50 mm × 2.5 mm × 0.2 mm i.d.) was used.

Table 1
Instrumental and operating conditions for ICP-AES measurements.

Parameter	Type or amount
Nebulizer	Concentric glass
Spray chamber	Cyclonic
RF power (kW)	1.15
Carrier gas (Ar) flow rate (L min^{-1})	0.6
Auxiliary gas (Ar) flow rate (L min^{-1})	1.0
Coolant gas (Ar) flow rate (L min^{-1})	14
Nebulizer flow (psi)	30
Pump rate (rpm)	100
Observation height (mm)	15
Integration time (s)	
On-axis	20
Off-axis	5
Wavelength (nm)	Sm(III) 442.434 nm

2.3. Sample preparation

The reference material (GBW07602, shrubbery leaves) was obtained from National Research Center for Certified Reference Materials (Beijing, China). Waste water sample containing REEs was collected from Jin chuan group Ltd. The reference material (GBW07602) was digested according to the literature [15]. Leaves were dried to constant weight in an oven. 1.0 g accurately weighed leave sample was transferred into a digestion tube, and then 5 mL of concentration HNO_3 was added into it. The tube was placed in a digester block and heated slowly until the temperature was up to 165 °C before it was left over night at room temperature. The tube was cooled down, and then 1.3 mL perchloric acid was added into it. Then the temperature was raised to 210 °C until evolution of white fume began. After the tube was cooled down, the volume was adjusted to 100 mL with double distilled water.

2.4. Preparation of new material

BT was activated through dry process: 10% HCl (v/v) was added to produce abundant silicone hydroxyl and aluminum hydroxyl, then filtered and repeatedly washed with water until the filtrate is neutral and dried in an oven at 160 °C for 8 h to remove surface-adsorbed water. 10.0 g activated BT was suspended in 150 mL dry toluene containing 10 mL of WD-30 and refluxed 12 h. The product (BT-CPTS) was BT bound chloropropyl and it was filtered off, washed with toluene, ethanol (95%) and dried in an oven at 80 °C for 6 h.

N-(2-Hydroxyethyl) ethylenediamine to modify activated bentonite was accomplished as following procedure: 5.0 g BT-CPTS reacted with 25 mL NHED in 250 mL flask, 100 mL of 95% ethanol as solvent. They were refluxed for 24 h, and then were filtered off, washed with ethanol (95%) and ether and dried in an oven at 80 °C for 6 h. This is the final product named BT-NHED and the synthetic route is illustrated in Fig. 1.

2.5. Procedures

2.5.1. Batch method

The experiment about the application of the new material was repeated eight times to ensure that there is satisfactory statistically evaluation.

A series of standards or sample solutions containing Sm(III) were transferred into a 25 mL beaker, 0.1 M HCl or 0.1 M $NH_3{\cdot}H_2O$ was used to adjust to the target pH value. Then the volume was adjusted to 10 mL with double distilled water of corresponding acidity. 30 mg of BT-NHED was subsequently added into the solution, the mixture was shaken vigorously for 30 min to facilitate adsorption of Sm(III) ions onto the sorbents. After extraction, the concentrations of Sm(III) ions in solution were directly determined by ICP-AES.

2.5.2. Column method

Right amount of BT-NHED was packed in a glass micro-column which was plugged with a small portion of glass wool at both ends. Prior to use, 0.5 mol L^{-1} HCl and double distilled water were successively passed through the column in order to equilibrate, clean and neutralize it. Each solution containing 1.0 $\mu g\ mL^{-1}$ Sm(III) was

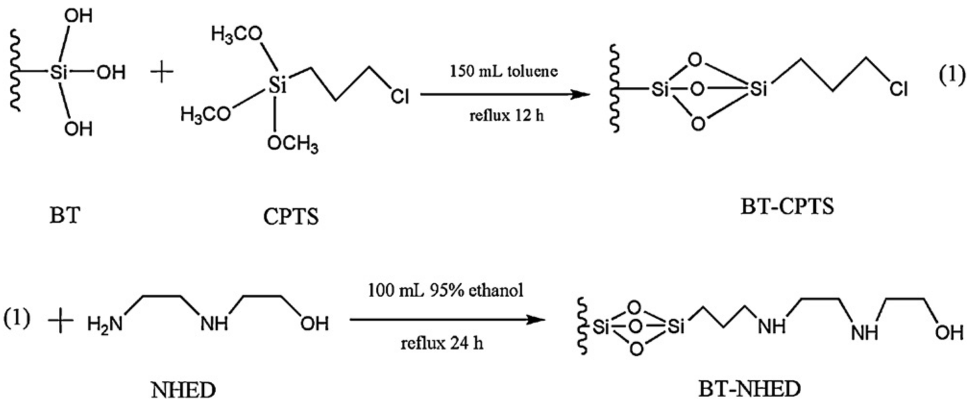

Fig. 1. Synthesis route of the BT-NHED.

passed through the column at the optimum flow rate controlled by a peristaltic pump. The bound Sm(III) ions were stripped off from the column with eluent. The analytes in the elution were determined by ICP-AES.

3. Results and discussion

3.1. Characterization of BT-NHED

The new material was characterized by several means: Fourier transform infrared (FT-IR, Fig. 2), thermogravimetry analysis (TGA, Fig. 3) and elemental analysis (as shown in Table 2).

Compared with that of BT-CPTS, the FT-IR of BT-NHED appeared several new peaks. According to the literature [16–18], 2953.75 cm^{-1} was due to the stretching vibration of intramolecular association of –OH of BT-NHED. The peak at 1459 cm^{-1} resulted from the deformation vibration of $-CH_2$ and at fingerprint region, 694.59 cm^{-1} was related to the non-plane rocking vibration of –NH. There are twin peaks at 1087.68 and 1041.05 cm^{-1}, which were stretching vibration of Si–O–Si, which can prove that the bentonite is Na substrate [19]. Besides, 3620.63 cm^{-1} and 793.27 cm^{-1} were attributed to stretching vibration of Al–OH and MgAl–OH, respectively. The peaks at 3426.13 and 1637.79 cm^{-1} were assigned to the stretching vibration of interlayer water molecules and bending vibration of H_2O. In fingerprint region, 520.11 cm^{-1} and 467.89 cm^{-1} were caused by Si–O–Mg and Si–O–Fe, respectively.

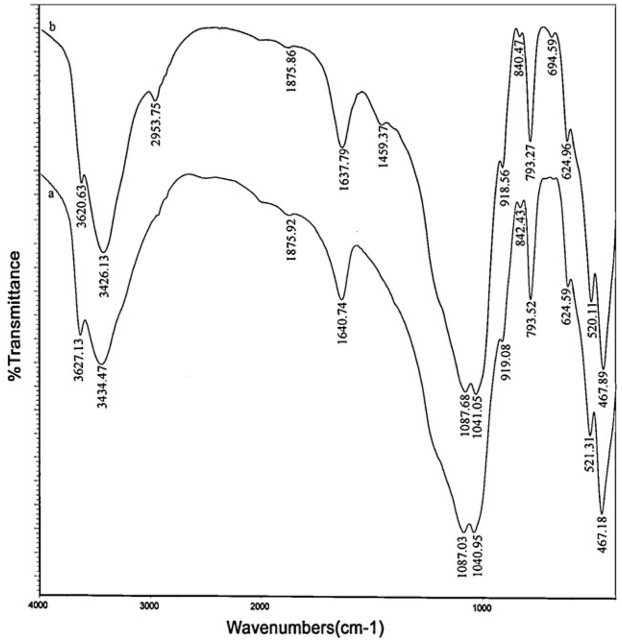

Fig. 2. FT-IR spectra of BT-CPTS (a) and BT-NHED (b).

Table 2
This is the data of elemental analysis.

Name	N (%)	C (%)	H (%)
BT	0.00	3.84	1.569
BT-NHED	2.16	8.19	1.711

Through observing the thermogravimetry (TG) curves of BT and BT-NHED presented in Fig. 3, several differences could be found between the two curves. Before 100 °C, weight percentage of BT and BT-NHED decreased 12% and 3%, respectively, which due to the loss of water. Ranged from 100 to 600 °C, the downtrend of weight percentage of BT changed slowly, however, there was about 11% weight of BT-NHED disappeared. The disappeared one in this range should be modifier (*N*-(2-hydroxyethyl) ethylenediamine). Higher than 600 °C, the rate of the weight losses for BT and BT-NHED was lower (only 2% and 1%), maybe the weight losses are the structure of bentonite changed [19]. All of the results confirmed that *N*-(2-hydroxyethyl) ethylenediamine modified bentonite successfully.

From the results of elemental analysis, a significant trend (0.00–2.16%) could be observed that the weight percentage content of N increases visibly. By calculating, the percentage of *N*-(2-Hydroxyethyl) ethylenediamine modified onto bentonite is 0.186 $g\,g^{-1}$. The results confirmed that bentonite was modified successfully and they were also consistent with above FT-IR and TG analysis results.

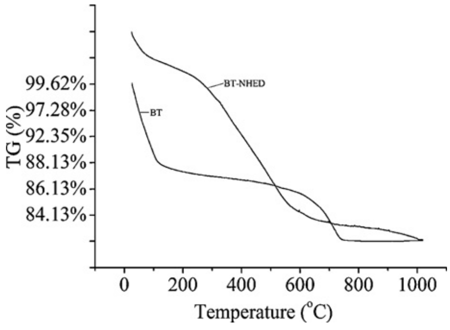

Fig. 3. TG curves of BT and BT-NHED.

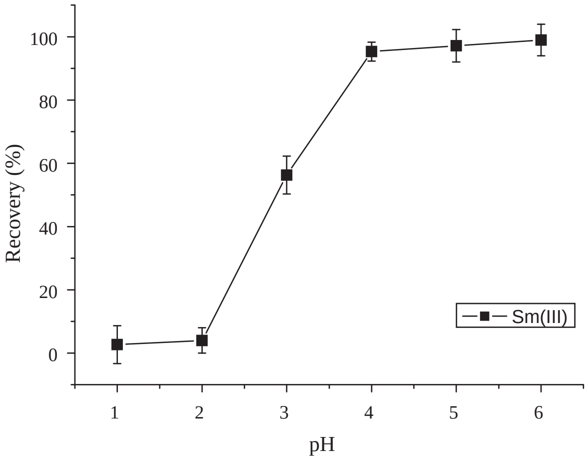

Fig. 4. Effect of pH on analyte adsorption; Sm(III): 1.0 μg mL^{-1}; sample volume: 10 mL; BT-NHED amount: 30 mg; shaking time: 30 min; temperature: 20 °C.

3.2. *Effect of pH*

The pH of solution has been known as the most important variable governing ions adsorption onto adsorbent. This is partly because hydrogen ions themselves are strongly competing with adsorbents [20]. According to the batch method, a series of 10 mL aliquots solution containing Sc(III), La(III), Er(III), Ce(III), Ho(III), Nd(III), Dy(III), Eu(III), Sm(III), Yb(III) were tested and determined by equilibrating with 30 mg sorbent at different pH values (range from 1 to 6). It could be seen from Fig. 4 that the optimum pH for quantitative extraction (>95%) of Sm(III) occurred at pH $\geq$ 4. In order to avoid metal precipitation experiments (pH values above 6) were not carried out. So, pH 4 was selected as the enrichment acidity for the rest of the experiment. In addition, at pH 4, Sc(III), La(III), Er(III), Ce(III), Ho(III), Nd(III), Dy(III), Eu(III), Yb(III) could be adsorbed by BT-NHED about 50–70%, but they do not interfere with enrichment and determination of Sm(III). At a low pH the surface of bentonite is more protonate, it is less able to retain ions [21]. The reason for this is that the surface complexation reactions are influenced by the electrostatic attraction between negatively charged groups at the bentonite surface and the ions [22].

3.3. *Effect of sorbent mass*

In the column procedure, appropriate amount of sorbent should be used in order to obtain quantitative retention of metals. Excess amount of the sorbent would slowed down the flow rate at a certain extent. The experiments of the sorbent mass were also investigated at pH 4. The result in Fig. 5 showed that quantitative recovery for Sm(III) was obtained in the range of 10–30 mg. To save the amount of the sorbent and improve the flow rate as far as possible, the column was filled with 10 mg BT-NHED in the further experiments.

3.4. *Effect of the shaking time*

A few minutes were needed to complete the Sm(III) ion extraction processes and it is another important factor in evaluating the affinity of BT-NHED to Sm(III). In this work, different shaking time (range from 2 to 30 min) was studied for the percentage extraction of Sm(III) by BT-NHED. The results showed that the adsorption of Sm(III) was over 95% during the first 2 min, which indicated that the new material possesses fast kinetics of adsorption equilibrium.

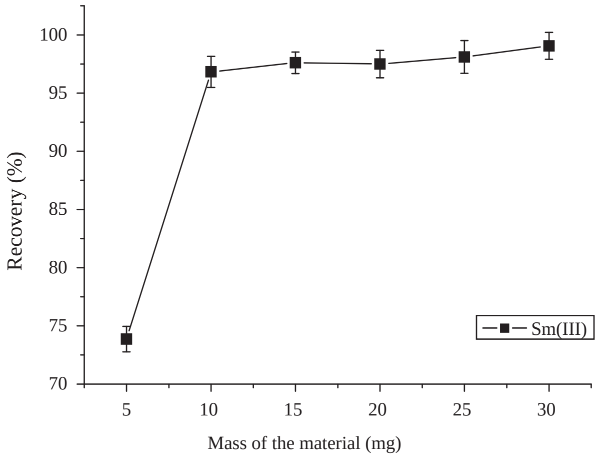

Fig. 5. Effect of BT-NHED amount on analyte adsorption; Sm(III): 1.0 μg mL^{-1}; sample volume: 10 mL; pH 4; flow rate: 0.5 mL min^{-1}; temperature: 20 °C.

3.5. *Effect of flow rate*

We investigated this factor using column procedure. Flow rate affects the retention of cations on the sorbent and the duration of complete analysis [23]. Under the optimum conditions (pH 4, sorbent 10 mg), 10 mL of sample solution containing Sm(III) was passed through the column. The flow rate was adjusted in the range of 0.5–4.0 mL min^{-1} with a peristaltic pump. As shown in Fig. 6 the recoveries of the studied ions decrease with the increase of the flow rate. In order to save time and guarantee quantitative recovery of Sm(III), the flow rate 1.0 mL min^{-1} was selected as the best one.

3.6. *Elution condition, maximum sample volume, enrichment factor and regeneration*

In order to realize repeated usage of the adsorbent column, elution condition has to be examined. The elution of Sm(III) from BT-NHED was examined by using 2 mL of various concentration of HCl or HCl + CS $(NH_2)_2$ eluent in the column experiment. The results listed in Table 3 showed that the recovery was about 60% using HCl as eluent. However, HCl + CS $(NH_2)_2$ were sufficient for stripping off the adsorbed Sm(III). The reason may be that thiourea and Sm(III) had stronger coordinating ability than that of BT-NHED, so Sm(III) ions were easily released from BT-NHED into the desorption medium. Therefore, the quantitative recovery of Sm(III) could

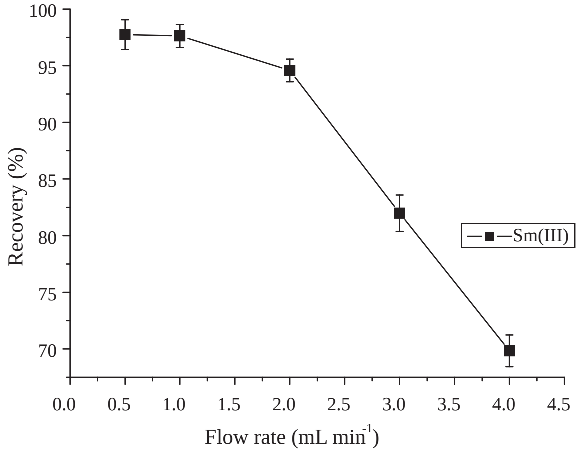

Fig. 6. Effect of flow rate on analyte adsorption; Sm(III): 1.0 μg mL^{-1}; sample volume: 10 mL; pH 4; BT-NHED amount: 10 mg; temperature: 20 °C.

Table 3
Eluent recovery (%) for Sm(III) adsorbed on BT-NHED.

Eluent	Recovery (%)
0.1 M HCl	69.28
0.5 M HCl	68.60
1 M HCl	67.97
2 M HCl	67.15
0.1 M HCl + 3% $CS(NH_2)_2$	89.43
0.1 M HCl + 4% $CS(NH_2)_2$	94.22
0.1 M HCl + 5% $CS(NH_2)_2$	96.83
0.5 M HCl + 3% $CS(NH_2)_2$	88.83
0.5 M HCl + 4% $CS(NH_2)_2$	87.46
0.5 M HCl + 5% $CS(NH_2)_2$	94.08

be obtained using 2 mL 0.1 M HCl + 5% CS $(NH_2)_2$ (% w/w) solution as eluent.

In order to achieve a high preconcentration factor, Sm(III) in the large sample volume has been quantitatively adsorbed and desorbed by a small stripping volume [24]. 50, 100, 150, 200, 250 and 300 mL of 1 μg mL^{-1} of Sm(III) were passed through the column padded with 10 mg sorbent at optimum flow rate. Quantitative recoveries (>95%) of Sm(III) was obtained when the sample volume less than 150 mL, and the percent of recoveries decreased at higher volume. Thus, 150 mL was adopted as the maximum sample volume for the preconcentration of samarium from sample solution. The enrichment factor was 75 because 2 mL eluent was used in these experiments.

Regeneration was also investigated as follows: at optimum flow rate, the solution containing Sm(III) (1 μg mL^{-1}) was passed through the column which filled with 10 mg sorbent and glass wool. After the adsorbed process finished, 2.0 mL 0.1 mol mL^{-1} HCl and 5% (% w/w) thiourea penetrated the column and the eluent was measured directly by ICP-AES. When it repeated 6 times, the recoveries of Sm(III) still reached 95%. Over 6 times, the recovery decreased. So, the repeat times of usage of the column are 6 times.

3.7. *Adsorption capacities*

The capacity study was adopted from the paper recommended by Maquieira et al. [25]. 30 mg of sorbent was equilibrated with 10 mL of various concentrations (50–300 μg mL^{-1}) of Sm(III) at pH 4 for 24 h in order to get "saturation". According to the equation (1) [6], we can calculate that the maximum adsorption capacity was 17.7 mg g^{-1} for Sm(III).

$$q = (C_0 - C_e)V/m, \qquad (1)$$

where q represents the adsorption capacity (mg g^{-1}), C_0 and C_e represent the initial and equilibrium concentration of Sm(III) (μg mL^{-1}), m is the mass of BT-NHED (g) and V is the volume of sample solution (mL)

3.8. *Effect of coexisting ions*

Rare earth elements are usually symbiotic and their chemical properties are similar. So, no matter which rare earth elements interfere into the assay of Sm(III) should be investigated. 1.0 μg mL^{-1} of Sm(III) containing the added interfering ions was prepared, according to the batch procedure. The tolerance limit was set as the amount of ions causing recoveries of the examined elements to be less than 90%. As shown in Table 4 that 2000 μg mL^{-1} K^+, Na^+, NO_3^-, Cl^-, 500 μg mL^{-1} Ca^{2+}, Mg^{2+}, HPO_4^{2-}, 50 μg mL^{-1} Er^{3+}, Dy^{3+}, Eu^{3+} and Fe^{3+}, 30 μg mL^{-1} Nd^{3+}, Ho^{3+}, Ce^{3+}, Ce^{4+}, Yb^{3+}, La^{3+} and Cr^{3+}, 20 μg mL^{-1} Sc^{3+}, Y^{3+} and Al^{3+} did not significantly interfere with the determination of the analytes under the selected conditions.

Table 4
Effect of coexisting ions on recovery of 1.0 μg mL^{-1} Sm(III) adsorbed on BT-NHED.

Coexisting ions	Concentration (μg mL^{-1})	Recovery (%)
Na^+	2000	98.69
K^+	2000	97.66
Cl^-	2000	96.53
NO_3^-	2000	95.88
Ca^{2+}	500	98.70
Mg^{2+}	500	94.50
HPO_4^{2-}	500	95.31
Er^{3+}	50	95.58
Eu^{3+}	50	92.54
Dy^{3+}	50	96.89
Nd^{3+}	30	93.56
Ho^{3+}	30	91.23
Ce^{3+}	30	95.35
Ce^{4+}	30	95.12
Yb^{3+}	30	93.03
La^{3+}	30	92.16
Sc^{3+}	20	90.31
Y^{3+}	20	90.02
Al^{3+}	20	91.56
Fe^{3+}	50	98.97
Cr^{3+}	30	96.45

Table 5
Analytical results for the determination of Sm(III) in certified reference materials and environmental samples.

Sample	Concentration of Sm(III) (μg g^{-1})				Recovery (%)
	Certified	Found	Added	Sum	
GBW07602	0.19 ± 0.01	0.18 ± 0.01[a]			
Waste water sample		0.71 ± 0.05	5.0	5.63 ± 0.13	98.40
			10.0	10.59 ± 0.17	98.80

[a] $\bar{x} + s$ ($n = 8$). $\bar{x}$ is average value for eight determinations and s is standard deviation.

3.9. *Detection limit and analytical precision*

Under the selected conditions, eight portions of standard solutions were analyzed following the batch procedure. The detection limit of the method was calculated based on three times of the standard deviation of eight runs of the blank solution [26]. The detection limit (3σ) was found to be 0.60 ng mL^{-1} for Sm(III). The relative standard deviation (RSD) of the eight replicate determinations was 2.6%. The results indicated that the method exhibited low detection limit and good precision for the analysis of Sm(III) in solution samples.

3.10. *Application of the method*

All of the optimized parameters were used for real samples to test the accuracy of the method.

This method has been applied to determine trace Sm(III) in certified reference material (GBW07602) and waste water sample. The results listed in Table 5. Good uniformity was obtained by the proposed method and the certified values of Sm(III). In waste water sample, the standard addition method was used. The analytical results showed that the recoveries of Sm(III) were reasonable for trace analysis. It demonstrated that the method was appropriate for the preconcentration of Sm(III) at the trace level in environmental samples.

4. Conclusions

This is the first time bentonite was treated in this way and modified as a SPE sorbent. FT-IR, elemental analysis and thermogravimetric (TG) were used to characterize BT-NHED successfully.

In SPE method, by using batch and column procedure, the new material exhibits excellent adsorptive selectivity towards Sm(III) in sample solution. Fast adsorption equilibrium, easy elution and good adsorption capacity were its other good characteristics. Moreover, the new material (BT-NHED) has been successfully applied to the analysis of trace Sm(III) in environmental samples. In summary, BT-NHED has higher analytical potential for preconcentration of trace Sm(III) from authentic samples.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.talanta.2010.12.012.

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