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A novel sampling strategy for metal ions determination in water: application to the determination of cadmium

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ABSTRACT

This work proposes a novel, portable, and reusable device for in situ water sampling, sample preparation, and analyte enrichment in a single step. For this purpose, a commercial cationic exchange resin (Chelex 100), was used to obtain a solid-phase extraction column for cadmium (II) retention and enrichment. The optimization of key parameters such as sample volume, reagents concentration, and flow-rate, was performed to maximize cadmium retention and elution efficiency. The method was applied to real samples, and the results agreed with those obtained with reference methodologies, with no significant differences (< 10%). Additionally, recovery rates of 97.8 ± 3.5% were achieved. This method integrates, in one step, the sampling and sample preparation procedures and employs reusable devices, contributing to providing a sustainable solution for metal ions monitoring in aquatic environments.

1. Introduction

The content of some metal ions in aquatic ecosystems has emerged as one of the most concerning topics, posing significant risks to both aquatic ecosystems and human health [1]. In this context, metal ions such as lead, zinc, mercury, cadmium, copper, chromium, and arsenic are among the metals of most interest [2]. Even at low exposure levels, these metal ions can lead to serious health problems, including cancer, neurological disorders, and damage to vital organs [3]. The cadmium content in natural waters presents a growing concern due to its widespread use in electric batteries, electronic components, water heaters, coolers and plumbing [4]. Given the serious risks associated with this type of contamination, regular water quality monitoring is crucial for effective pollution control and risk management.

Traditional water sampling methods for metal analysis typically involve bottle sampling and the addition of preservatives before transportation to the laboratory, where samples are refrigerated until analysis. Additionally, at the lab, samples often undergo treatment steps to remove interferences or enrich the analyte before the quantification, usually performed by atomic absorption spectrometry (AAS) or inductively coupled plasma - atomic emission spectrometry (ICP-OES) [5]. Although this sampling procedure has been widely used for several years, it presents several challenges/limitations that can affect the quantification accuracy. Some issues may arise during bottle sampling and transportation to the laboratory, as samples may be susceptible to degradation. This can occur due to temperature variation, UV irradiation, and microbial activity, among others, modifying the initial composition of the sample and possibly affecting some natural equilibria [6,7]. Therefore, the integrity of the sample can be compromised, introducing uncertainty in the quantification. Additionally, during sample pre-treatment, in which processes like liquid-liquid extraction and liquid-solid phase extraction are performed, the sample composition can also be further affected. And, again, factors such as oxygen exposure, changes in pH and the use of organic solvents can impact the integrity of the sample [8,9]. Besides the potential sample alteration, these processes may increase the time required for analysis and the complexity of the process.

This work proposes a novel method for water sampling based on solid-phase extraction. The main goal is to integrate sampling and pretreatment of the samples in a single step for cadmium quantification in freshwater. To achieve this, a portable and reusable device using a commercial cationic exchange resin (Chelex 100) was devised. Chelex 100 is a styrene divinylbenzene copolymer that contains paired iminodiacetic ions, thus acting as a chelating ion exchange resin with high selectivity for metal ions [10]. These characteristics make it particularly interesting for retaining metal ions from water and allowing the

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possibility to eliminate most of the water matrix. Additionally, once the selectivity of the resin can be controlled through pH, the elution of the metal ions can also be controlled [11]. Moreover, advantage was taken from the resin features to enrich the analyte, as the cadmium levels in waters are usually between 3 and 5 μg L^{-1}, which is often below the limit of detection of several traditional methods [12].

The objective was to establish a miniaturized extraction procedure able to retain metal ions at the source for subsequent transport to the laboratory for further quantification. This procedure could be an advantageous alternative to the conventional transport and storage of a high volume of water samples from the sampling site to the lab. The development of the proposed water sampling strategy involved a two-stage optimization process to ensure the best performance of the method, namely at the retention and elution steps. The influence of several physical and chemical parameters was evaluated to maximize cadmium retention, including the sampling volume and the NaOH concentration used for column conditioning. The concentration and volume of the elution solution (HNO_3), as well as the elution flow-rate, were optimized to enhance cadmium elution. The final conditions were set as those providing the highest retention capacity and elution efficiency (≈ 100 %). The method was validated by comparing the results obtained from the newly developed strategy with those from the reference, demonstrating no significant differences (< 10 %).

2. Material and methods

2.1. Reagents and solutions

All solutions were prepared with analytical grade chemicals and MilliQ water (resistivity > 18MΩcm, Millipore, Burlington, MA, USA).

A stock solution of 5 mg L^{-1} of cadmium was prepared by dilution of the respective atomic absorption standard solution of 1000 mg L^{-1} (Merck KGaA, Darmstadt, Germany). Working standard solutions in the range of 0.005–0.50 mg L^{-1} were prepared weekly by dilution of the 5 mg L^{-1} stock solution and acidified to a final concentration of 0.1 mol L^{-1} nitric acid.

The 0.1 and 0.5 mol L^{-1} nitric acid solutions were prepared by dilution of the commercial concentrated nitric acid solution ($d = 1.39$, 65 %, Supelco, USA).

A 0.5 mol L^{-1} sodium hydroxide stock solution was prepared by dissolving 10 g of sodium hydroxide (PanReac, Spain) in 500 mL of water.

Before the cadmium atomic absorption spectrometry quantification, a solution of lanthanum (III) 100,000 mg L^{-1} was added to the sample to a final concentration of 4000 mg L^{-1}.

An acetate buffer solution was prepared by dissolving 6.56 g of sodium acetate (Merck, Germany) in 16 mL of 100 % acetic acid (Merck, Germany) and then diluted to a final volume of 200 mL with water (pH 4).

2.2. Preparation of the sampling device

For the sampling procedure, a sampling device (SD) was prepared. For that, a laboratory-made column was made using 1 cm of Tygon tubing (Gilson, Villiers-le-Bel, France) with an inner diameter of 3.18 mm and an internal volume of 80 μL (Fig. 1). Chelex 100 resin (mesh size 50–100, Bio-Rad, USA) was suspended in acetate buffer solution. Approximately 70 mg of the resin was packed into the column and secured between two pieces of dishwashing sponge. Before the sampling procedure, the column was conditioned with 1 mL of NaOH 0.50 mol L^{-1}. This step was followed by the column rinse with 5 mL of deionised water.

2.3. Flow-based setup

Fig. 1. Photograph of the Chelex 100 packed column alongside a one-euro coin for scale.

A flow-based setup was assembled resorting to a Crison MultiBurette BU 4 S (Crison, Barcelona, Spain), equipped with two glass syringes, a 5 mL Gastight Syringe Model 1005 TLL, PTFE Luer Lock Part (Hamilton Company, USA) and a 10 mL Gastight Syringe Model 1010 TLL, PTFE Luer Lock Part (Hamilton Company, USA), to propel HNO_3 0.5 mol L^{-1} and deionised water, respectively. Connected to each syringe were two PTFE tubes, 1/16″ (1.6 mm) OD x 0.8 mm ID (Diba Omnifit, United Kingdom), one submerged in the solution to fill the syringe and the second one to connect to the sampling device. This setup performed both the conditioning of the column and the analyte elution.

2.4. Water sampling procedure

For water sampling, a 5 mL Injekt® luer solo syringe (B. Braun, Germany) was filled with standard/sample and connected to one end of the SD (Chelex-100 packed column). Afterwards, the standard/sample was passed through the column manually, aiming for a flow rate of approximately 1 mL min^{-1}. Once the sample passes through the column, the column was connected to the multi-burette, and an additional 1 mL of water was perfused through the SD at the same flow rate to ensure all sample volume is flushed from the column (this step can also be performed manually in situ). This solution was collected for further analysis.

After the retention of the metal ions from the sample in the SD, the analyte elution was performed. The column was attached to the multi-burette syringe containing HNO_3 0.5 mol L^{-1}, and 1 mL was dispensed through the column at 2 mL min^{-1}. To ensure complete elution, an additional 1 mL of MQW was injected at the same flow-rate to flush the remaining cadmium ions from the column. This step introduces a two-fold dilution factor to the sample while simultaneously achieving a 5-fold preconcentration, as only 1 mL of eluent is collected. The eluted solutions were collected for subsequent analysis, and lanthanum was added before quantification to a concentration of 4000 mg L^{-1}. The elution step was repeated once more to ensure the clean-up of the column to reuse the SD. The full procedure, from the column conditioning until the analyte elution for the cadmium quantification, is summarised in Table 1.

2.5. Cadmium quantification

The eluted samples were analyzed resorting to atomic absorption spectrometry (AAS) with an Unicam AAS 969 spectrometer (Solaar, Germany). For the analysis, the spectrophotometer was equipped with a cadmium hollow cathode lamp 37 mm (Kinesis, United Kingdom). Each solution, obtained from both sampling and elution, was analyzed in triplicate. The cadmium concentration ($[Cd^{2+}]_{AAS}$) was then determined

Table 1
Procedure for the sampling device preparation/conditioning and its use for cadmium collection, elution, and quantification.

Procedure	Reagents and conditions	Purpose/description
Column conditioning	1.0 mL of NaOH 0.50 mol L^{-1}	Increase the retention capacity of Chelex 100.
	5.0 mL of water	Rinse the excess of NaOH from the column.
Sampling	5.0 mL of sample	Perfusion of the sample through the column for retention of metal ions.
	1.0 mL of water	Ensure the discharge of the sample matrix.
Elution	1.0 mL of HNO_3 0.50 mol L^{-1} at 2.0 mL min^{-1}	Elution of the metal ions from the column.
	1.0 mL of water	Ensure the complete elution of the metal ions.
Quantification*	80 μL of lanthanum 100,000 mg L^{-1}	Reducing interferences.

* Quantification performed by atomic absorption spectrometry.

based on a calibration curve previously established with cadmium standards ranging from 0.005 to 0.50 mg L^{-1}. To calculate the cadmium concentration of the sample ($[Cd^{2+}]$), parameters as the dilution (2 times) and concentration (5 times) factors had to be taken into account, DF and CF, respectively. This can be resumed by the following equation:

$$[Cd^{2+}] = \frac{[Cd^{2+}]_{AAS} * DF}{CF}$$

DF – $Dilution\ factor\ (2x)$

CF – $Concentration\ factor\ (5x)$

3. Results and discussion

3.1. Preliminary studies

Considering one of the main objectives of the herein proposed method, which was to simplify the overall sampling procedure and sample preparation, it was crucial that it could be performed in situ. To achieve this, traditional plastic syringes were tested to collect the sample and conduct the sampling right afterwards. The uncertainty of the volumes measured with this kind of syringe was assessed. Thus, syringes with different inner volumes were tested (2, 5, 10, and 20 mL). By using the different syringes at the respective highest capacity, it was possible to determine from 10 measurements that, for all the syringes tested, the error was below 1 %.

3.2. Optimization of the cadmium retention

The main goal of the proposed sampling method was to develop a device packed with Chelex 100, a cation exchange, for water sampling, enabling the analyte retention and matrix removal within a single in situ step. Since Chelex 100 is a non-selective sorbent material, some studies were performed to maximize the cadmium retention within the packed column. This was accomplished by injecting a cadmium solution with a known concentration (100 μg L^{-1}) into the column and, after that, analyzing the solution that perfused through the column. The objective was to determine the SD retention capacity for cadmium ions. Two key parameters were evaluated and optimized: the volume of the sample injected and the NaOH concentration used for column conditioning.

Firstly, the optimization of the retention was conducted without pre-conditioning the column. For that, 2 mL, 5 mL and 10 mL of a 100 μg L^{-1} cadmium standard solution were injected into the SD. The results indicated incomplete cadmium retention across all injected volumes, with no significant differences for the 2 mL and 5 mL samples (Fig. 2A). To improve the retention capacity, the SD was conditioned with 1 mL of a NaOH solution with 3 different concentrations (0.01, 0.1, and 0.5 mol L^{-1}), before the standard solution perfusion. After conditioning, 5 mL of a 100 μg L^{-1} cadmium standard solution was injected, and the retention capacity increased as the NaOH concentration increased, achieving a 100.0 ± 0.1 % retention with the 0.5 mol L^{-1} of NaOH solution (Fig. 2B). All further experiments employed the pre-conditioning of the column with 1 mL of a 0.5 mol L^{-1} NaOH solution, combined with a sample injection volume of 5 mL.

3.3. Optimization of the cadmium ions elution

The next step for the method development involved optimizing the elution of the cadmium ions from the column. Several parameters were assessed, namely the concentration and volume of the elution solution (HNO_3), and the elution flow-rate.

Initially, the volume and elution flow-rate of HNO_3 were set at 1 mL and 1 mL min^{-1}, respectively, while varying its concentration (Fig. 3A). The tested HNO_3 solution concentrations were 0.50, 0.25 and 0.10 mol L^{-1}. Using a 0.5 mol L^{-1} HNO_3 solution, an elution of 106.5 ± 0.0 % of cadmium ions was achieved. Then, a higher elution flow-rate was tested to reduce the overall method time (Fig. 3B). Elution flow-rates of 1.0, 2.0, and 3.0 mL min^{-1} were tested. It was found that a rate of 2.0 mL min^{-1} maintained elution efficiency, with 102.0 ± 2.6 % of eluted cadmium. Different volumes of HNO_3 for the elution process were also considered, but since approximately 100 % was consistently achieved with 1 mL, this volume was maintained. As a result, the parameters set for the elution process were 1.00 mL of 0.50 mol L^{-1} HNO_3 at a flow-rate

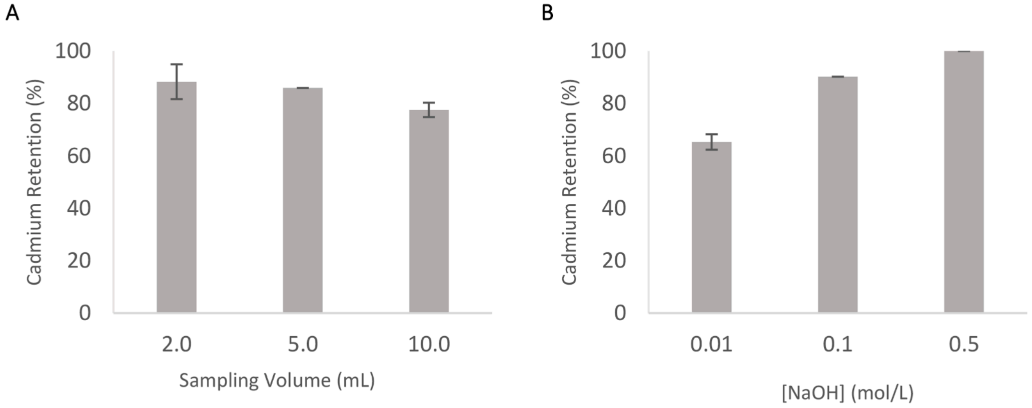

Fig. 2. Retention capacity optimization of the sampling device, packed with Chelex 100, for cadmium quantification. A) Percentage of cadmium ions retained with different sample injection volumes, without column pre-conditioning. B) Percentage of cadmium ions retained after column pre-conditioning with different NaOH concentrations.

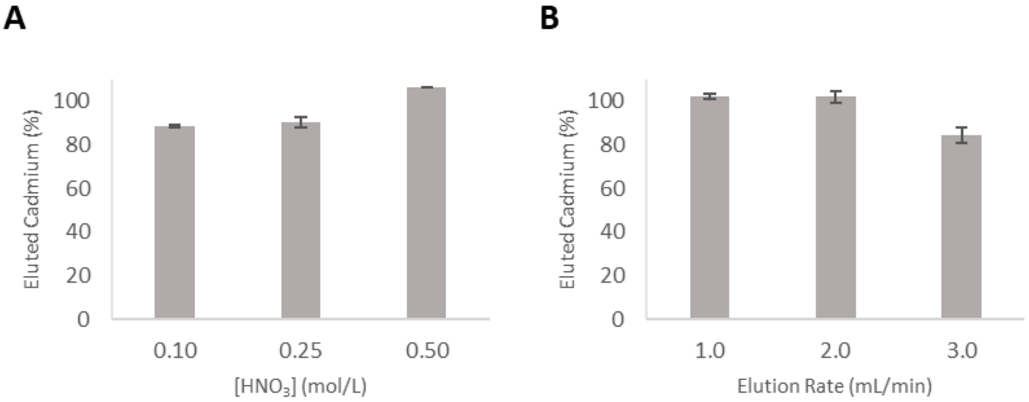

Fig. 3. Cadmium elution optimization studies from a sampling device packed with Chelex 100. A) Percentage of cadmium ions eluted with different HNO_3 concentrations. B) Percentage of cadmium ions eluted varying elution flow-rate.

of 2.0 mL min^{-1}.

3.4. Interference assessment

The developed sampling device uses a commercially available sorbent material, Chelex 100, that exhibits affinity towards various metal ions. This general affinity could potentially compromise both retention and elution of cadmium. Therefore, the potential interference of other metal ions that can be present in freshwater was evaluated within the proposed sampling strategy for cadmium quantification. For that, 100 μg L^{-1} cadmium standard solutions were prepared, and different metal ions were added in concentrations normally present in this type of water [13]. The interference was assessed at both the retention and elution steps. No significant interferences were observed in the retention step, as retention of cadmium ions achieved nearly 100 % (cadmium concentration lower than the limit of detection in the solutions that perfused the SD), regardless of the presence of interferents (Table 2).

The interference of the metal ions was also assessed in the eluted solutions. As water samples typically contain high quantities of calcium, which can form calcium pyrophosphate and interfere with the analysis, a solution of lanthanum was added to the eluted solution to act as a releasing agent during the AAS quantification procedure. For that, 80 μL of the lanthanum solution was added to each solution volume to achieve a final concentration of 4000 mg L^{-1}. The results (Table 3) also showed that the presence of other metal ions did not significantly interfere ($<$ 10 %) with the cadmium elution and quantification.

3.5. Method validation

Method validation was carried out by analysing various water samples using both the novel method and a reference procedure (the samples were analysed directly through AAS), followed by a comparison of the results. The results are depicted in Table 4.

Table 2
Assessment of potential interference of various metal ions in cadmium retention in the column; each assay was performed with a standard solution of cadmium 100 μg L^{-1}. Cadmium concentration was assessed according to the calculation demonstrated in Section 2.5.

Metal ion	$[Me]_{streams}$ (mg L^{-1})	$[Me]_{groundwaters}$ (mg L^{-1})	$[Me]_{added}$ (mg L^{-1})	$[Cd]_{found}$ ± StD (mg L^{-1})
Al^{3+}	0.4	$<$ 0.0001	0.4	<LOD
Ca^{2+}	15.0	1 – 500	15.0	<LOD
Co^{2+}	0.0003	0.001 – 0.01	0.0100	<LOD
Cu^{2+}	$<$ 0.012	$<$ 0.01	0.100	<LOD
Mg^{2+}	4.0	$>$ 5.0	5.00	<LOD
Mn^{2+}	0.007	$<$ 0.10	0.100	<LOD
Ni^{2+}	0.001	$<$ 0.10	0.100	<LOD
Zn^{2+}	0.02	$<$ 0.10	0.100	<LOD

[Me], metal ion concentration; StD, standard deviation.

A linear relationship between the results of the reference method (AAS) and the novel method (NM) was established, and the equation was NM = (0.98 ± 0.12)AAS – (2.4 ± 8.3). The values in parentheses are 95 % confidence limits. This shows that the estimated slope and intercept do not differ from the values 1 and 0, respectively. Therefore, there is no evidence for systematic differences between the two sets of results [14].

Additionally, for validation purposes, recovery experiments were conducted. In these experiments, four water samples were spiked with known cadmium concentrations and analyzed both before and after spiking to assess the method's accuracy.

The recovery percentage calculations were made according to the IUPAC recommendations and the results are depicted in Table 5 [15]. The developed method provided recovery ratios of 97.8 ± 3.5 %. The statistical *t*-test for a 95 % significance level was calculated. The obtained t-value was 0.406 and the corresponding critical value was 2.776, meaning there is no evidence of systematic errors or matrix interference [16].

The limits of detection and quantification, LOD and LOQ, were calculated according to IUPAC recommendations [17,18], being respectively three and ten times the standard deviation of ten consecutive measurements of a blank solution (deionised water). The results were a LOD of 5.0 μg L^{-1} and a LOQ of 13.4 μg L^{-1}.

3.6. Stability and reusability of the column

The reusability of the Chelex-packed column was evaluated through two variables: the number of uses and the duration of use. The column was used for up to 50 consecutive sampling and elution cycles, and it was tested over a two-month period with intermittent use. In both cases, no significant loss in retention capacity or elution performance was observed, demonstrating the column's stability and durability.

4. Conclusions

In this work, a novel method for water sampling and sample preparation envisaging cadmium quantification in freshwater was developed using a sampling device packed with a commercially available resin (Chelex-100). Solid-phase extraction was the approach used to integrate, in a single step to perform in-situ, the sampling and pre-treatment of the water sample. As far as we know, the water sampling reference procedure [19] for metal ions quantification is still based on bottle sampling, sample acidification, transport to the laboratory, refrigeration, and the need to undergo sample treatment before analysis. The herein described approach successfully overcomes common issues found in traditional sampling methods and sample treatment procedures, such

Table 3
Assessment of potential interference, of various metal ions, in cadmium elution from the column. Each assay was performed with a 100 μg L^{-1} cadmium standard solution. Cadmium concentration was assessed according to the calculation demonstrated in Section 2.5.

Metal ion	$[Me]_{streams}$ (mg L^{-1})	$[Me]_{groundwaters}$ (mg L^{-1})	$[Me]_{added}$ (mg L^{-1})	$[Cd^{2+}]_{found}$ ± StD (mg L^{-1})	Interference (%)
Al^{3+}	0.4	< 0.0001	0.4	82.0 ± 5.9	- 18.9
			0.2	104.3 ± 1.3	+ 4.3
Ca^{2+}	15.0	1 – 500	15.0	83.9 ± 4.4	- 16.1
				98.2 ± 1.7	- 1.8
Co^{2+}	0.0003	0.001 – 0.01	0.0100	104.8 ± 2.6	+ 4.8
Cu^{2+}	< 0.012	< 0.01	0.100	92.2. ± 6.0	- 7.8
Fe^{3+}	0.7	0.10 - 10		80.1 ± 2.1	- 19.9
				108.3 ± 1.6	+ 8.3
Mg^{2+}	4.0	> 5.0	5.00	80.0 ± 2.1	- 20.0
				90.6 ± 4.7	- 9.4
Mn^{2+}	0.007	< 0.10	0.100	102.6 ± 7.2	+ 2.6
Ni^{2+}	0.001	< 0.10	0.100	106.1 ± 3.6	+ 6.1
Zn^{2+}	0.02	< 0.10	0.100	100.9 ± 0.2	+ 0.9

StD, standard deviation.

Table 4
Analysis of cadmium in different well water samples through the reference method (AAS) and the developed method. Cadmium concentration was assessed according to the calculation demonstrated in the Section 2.5.

Sample ID	Reference method (μg L^{-1})	Novel method ± StD (μg L^{-1})	RE (%)
#1	14.8 ± 0.7	12.6 ± 0.7	−15.0
#2	25.9 ± 1.3	23.7 ± 5.2	−8.6
#3	29.6 ± 1.5	26.7 ± 0.0	−10.0
#4	<LOD	<LOD	–
#5	29.6 ± 1.5	32.6 ± 3.0	+10.0
#6	<LOD	<LOD	–
#7	<LOD	<LOD	–
#8	59.3 ± 2.9	46.7 ± 0.7	−21.25
#9	91.6 ± 4.6	86.3 ± 4.3	−5.8
#10	138.0 ± 6.9	136.3 ± 6.8	−1.2

RE, relative error; LOD, limit of detection; StD standard deviation.

Table 5
Recovery percentages calculated from spiked well waters assessed with the developed method. Cadmium concentration was assessed according to the calculation demonstrated in Section 2.5.

Sample ID	$[Cd^{2+}]_{Initial}$ (μg L^{-1})	$[Cd^{2+}]_{Added}$ (μg L^{-1})	$[Cd^{2+}]_{Found}$ (μg L^{-1})	Recovery (%)
#11	<LOD	25.0	24.7 ± 0.8	98.8
#11	<LOD	50.0	45.4 ± 2.4	90.8
#12	<LOD	25.0	25.4 ± 2.5	101.6
#12	<LOD	50.0	50.0 ± 2.5	100.0

LOD, limit of detection.

as possible sample degradation and complex pre-treatment procedures, which often compromise quantification accuracy. With the newly developed strategy, sampling and sample treatment (analyte enrichment and matrix removal) were achieved in one easy in situ step using the designed device packed with Chelex 100.

The high retention and elution efficiency of the developed method enabled the enrichment of cadmium. Combined with a low LOQ (13.4 μg L^{-1}), this approach enabled precise cadmium quantification, even in samples with low concentrations.

This innovative method not only enhances analytical performance but also supports environmental sustainability, making it a valuable tool for real-time water quality monitoring and management of heavy metal pollution. Additionally, the ability to reuse the column for up to two months or 50 cycles further improves the method's cost-effectiveness and sustainability.

CRediT authorship contribution statement

Francisco A.P. Rodrigues: Writing – original draft, Investigation, Data curation. **Tânia C.F. Ribas:** Writing – review & editing, Validation, Supervision, Methodology, Investigation, Conceptualization. **Raquel B. R. Mesquita:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Conceptualization. **António O.S. S. Rangel:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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