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Synthesis of MIL101@ZIF67@ZIF8 Hybrid Nanostructure as a New Sorbent in Thin Film Microextraction for the Extraction and Measurement of Pb^{2+} and Cd^{2+} Metal Ions in Water and Cosmetics Samples by Flame Atomic Absorption Spectroscopy

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In this study, a new sorbent was synthesized based on the in-situ growth of the combined ZIF-67 and ZIF-8 on the surface of MIL101. Finally, the adsorbent was made into a thin film with the help of polyvinylidene difluoride polymer and was used in the TFME technique for the simultaneous measurement of cadmium(II) and lead(II) metal ions in aqueous and cosmetic samples using flame atomic absorption spectroscopy. In order to achieve maximum efficiency and sensitivity, parameters affecting the extraction and desorption processes were optimized. Under the optimized condition, the linearity of the method, the limit of detections (LODs), the limit of quantifications (LOQs), the relative standard deviations (RSDs%), and enrichment factors (EFs) were equal with 0.2-150.0 µg l^{-1}, 0.059-0.159 µg l^{-1}, 0.197-0.530 µg l^{-1}, 1.2 to 2.5% and 73-75, in water matrix, respectively. Then the optimal conditions for Pb and Cd were used in water and cosmetics samples by flame atomic absorption spectroscopy and acceptable results were obtained.

Keywords: Hybrid MIL101@ZIF8@ZIF67, TFME, Pb, Cd, Flame Atomic Absorption spectroscopy

INTRODUCTION

Cosmetic products are materials or products that are widely used as beauty products and skin protection against external stimuli [1]. These products are mainly used in contact with different parts of the human body, such as the epidermis, lips, eyes, hair, *etc*., and they affect not only the skin but also the health of the internal organs [2,3]. In addition to the required additives, cosmetics are generally composed of water, surfactants, polymers, oils, and metal powders. Some of the metals in the structure of cosmetics are used in the sample structure as needed. Some other metal powders such as lead (Pb) and cadmium (Cd) are unintentionally added as impurities to cosmetics as a result of release from metal devices used in the manufacture of

cosmetics during the production of a substance or a product [4,5]. In addition, lead metal may originate from some natural ingredients such as paint and pigments that contain lead [6, 7]. Lead and cadmium are among the pollutants found in different types of cosmetic products such as foundation creams, lipsticks, eyeliners, etc. According to recent studies, these metals (Cd, Pb) can cause different types of skin problems, as well as damage to different organs of the human body. Studies by the joint FAO/WHO committee indicate that the permissible amount of cadmium for a human is 7 µg kg^{-1} of body weight per week. Chronic lead poisoning can affect almost all body systems and a daily intake of 3.5 µg of lead per kilogram of body weight can cause chronic poisoning. Therefore, there are concerns about the presence of these heavy metals in cosmetic products [8].

The thin film microextraction method was presented for the first time by Pawliszyn and colleagues in 2003 AD [9].

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This method is considered one of the types of solid phase microextraction methods, which is widely used for sample preparation while being simple and cheap. In thin film microextraction, a thin membrane is made from commercially available laboratory materials, which are used as the extractant phase [10]. The membrane used in this method has a relatively large surface with very little thickness, so it increases the extraction efficiency without increasing the extraction time. In this technique, the used attractants are created in the form of a thin film on a completely flat surface. In the TFME method, a flat film plate or a completely smooth membrane with a high surface-to-volume ratio is used as the extractor phase. By using this method, the volume of the extractor phase increases, while the thickness of the extractor phase is constant or even decreases in some cases. In this case, the extraction efficiency increases [11]. Up to now, several different kinds of sorbents such as PAMAM-GO [12], ZIF8-GO [13], ZIF8-LDH-Cotton yarn [14], molecularly imprinted polymer (MIP) [15] and PDMS/DVB [16] have been synthesized and then employed to do the TFME.

Metal-organic frameworks (MOF) are composed of secondary structural units (for example, metal ions or metal clusters) and organic ligands. The MOFs obtained from the coordinated connection of polymers will form one-dimensional chains, two-dimensional layers, and finally three-dimensional networks. These frameworks are one of the most widely researched materials of the 21st century due to their proper structure, controlled porosity, and high crystallinity. Another advantage of these structures is having a high surface area, which varies from 1000 to 10000 m^2 g [17,18]. In addition, it is possible to change the MOF pore size and its shape by choosing different organic binders and changing the type of central metal [19]. Another advantage is that MOFs can be made on a large scale through low-cost and simple synthetic methods. In other words, metal-organic frameworks have emerged as an attractive subset of porous materials that simultaneously contain the characteristics of a natural crystal along with the advantages of ordinary polymers [20]. Imidazole zeolite frameworks (ZIFs) are a subset of emerging MOFs, which are composed of imidazole as a structural connecting unit and central cations such as cobalt and zinc as the central nuclei of this structure [21].

ZIFs have common advantages with MOFs, such as high surface area, regular and uniform distribution of holes, modifiability, and high thermal and chemical stability. In other words, imidazolium frameworks synthesized by doubly positive cations such as zinc and organic imidazolium linkers are known as one of the most common subsets of MOFs [22]. As mentioned before, in comparison with other MOFs, ZIFs not only have good thermal-chemical stability and water tolerance but also have adjustable topologies and an easy synthetic process to achieve uniform structures. Hybrid materials, especially nanostructures with high complexity, have attracted much attention due to their delicate structure and complex functions. An important family of porous crystalline materials is MOFs, whose special composition and structure are important factors for their use in various fields of science. To improve the complexity of MOF-based composites with broad functionalities, the assembly of MOFs and other nanomaterials is a promising approach. Also, combining two or more different MOFs into one MOF-on-MOF hybrid material provides another strategy to fabricate complex MOF hybrids with unprecedented nanostructures. To date, various structures of MOF-on-MOF hybrids have been developed, including core-shell [23], yolk-shell [24], asymmetric [25], and film-on-film structures [26]. However, current MOF-on-MOF systems are mainly focused on binary composites. Recently, the production of MOF-on-MOF ternary hybrids has been successful and the synthesis of MOFs with this method compared to the existing traditional MOFs leads to an increase in the contact surface between the adsorbent and the desired test species, thereby increasing the extraction efficiency and improving the performance of the method.

This research aims to synthesize a hybrid metal-organic framework with a very high effective surface area, consisting of MIL 101, ZIF-8, and ZIF-67. Finally, the adsorbent was made into a thin film with the help of polyvinylidene difluoride polymer and was used in the TFME technique for the simultaneous measurement of cadmium and lead metals in aqueous and cosmetic samples using flame atomic absorption spectroscopy. The effective parameters of this research were first identified using the Placket-Burman screening method, and then these effective parameters were optimized using the Box-Behnken design method.

EXPERIMENTAL

Materials, Instrumental, Methods and Preparation of Real Samples

The standards, reagents, and materials used in this research were all Merck and Sigma Aldrich products. Details about the devices used to identify the synthesized sorbent, the spectroscopic device used to measure the amount of metals, how to prepare standard solutions, the preparation of real samples, and so on are given in the supplementary information section.

Preparation of MIL 101, MIL101@ZIF67@ZIF8, and Thin Film

To prepare MIL-101(Fe) by solvothermal method, 4 mmol of $FeCl_3 \cdot 6H_2O$, 4 mmol of H_2BDC, and 1 ml of acetic acid are dissolved in 80 ml of DMF and stirred for 30 min at room temperature. Then the resulting mixture was transferred to a 100 ml Teflon-lined steel autoclave and kept at 120 °C for 20 h. Next, the resulting yellow solid was separated by centrifugation, washed with DMF, and dried at 60 °C. Next, for the synthesis of MIL 101@ZIF 67@ZIF8, 60 mg of MIL101 was dispersed in 3 ml of methanol, and then 50 ml of 80 mM 2-MIM and 30 ml of $Co(NO_3)_2 \cdot 6H_2O$ were added to the above mixture and stirred for 4 h at room temperature. Then the mixture was centrifuged, the resulting solid was separated and washed with methanol and dissolved in 5 ml of methanol. Then 2 ml of the resulting mixture was added to a container containing 30 ml of 25 mM 2-MIM and 50 ml of 25 mM $Zn(NO_3)_2 \cdot 6H_2O$ and stirred for 4 hours at room temperature. After separating from the mixture, the resulting solid was washed three times with methanol and dried at 60 °C. The details of the film production are given in the supplementary information section.

TFME Procedure

A 200 ml container was used for the extraction process. For this purpose, the prepared thin film was attached to the clip and mounted on the lid of the container. The container containing the extraction solution (200 ml of the test sample solution with a concentration of 50 μg l^{-1}, at which the solution pH was reached at 3.0 by the addition of an appropriate drop of HCl (1.0 M)) was placed on the stirrer and the thin film containing the absorbent was placed in the

solution. By turning on the stirrer, the extraction time of each stage is allowed to elapse for separation. Extraction was done at ambient temperature of the laboratory for 15 min at 1100 rpm. After this step, the thin film was removed from the container containing the extraction solution and allowed to dry. The container for the desorption stage was chosen in such a way that the thin film was completely placed in the desorption solvent. Now, the thin film is placed inside this container containing desorption solvent by a clean tweezer, and the time calculated for the desorption process is allowed to pass (5 min). To perform desorption, the container containing the desorption solvent was placed in the ultrasonic bath for the specified duration of the desorption stage. After the time of the desorption stage, the thin film was taken out of the solvent and washed with double distilled water, 2 or 3 times and then allowed to dry to be used for the next extraction solution. Finally, the preconcentrate detergent solution containing the examined metals was injected into the FAAS.

RESULTS AND DISCUSSION

Characterization of the MIL101 and MIL101@ZIF67@ZIF8

In the FT-IR spectrum related to MIL 101 Fig. 1A, the broad peak in the 1682 cm^{-1} absorption region can be attributed to C=O bonds in free carboxylic groups, indicating the dicarboxylate linker. Mil 101 shows peaks in the areas of 1583 cm^{-1}, 1396 cm^{-1}, and 750 cm^{-1}, which are related to asymmetric and symmetric vibrations of COOH and bending vibrations C-H of benzene. ZIF 67-8 have broad peaks in the range of 3435 cm^{-1} and 2929 cm^{-1}, which show the C-H stretching vibration. Also, the C=N stretching vibration of 2-MIM ligand and C-H bonds related to the aromatic ring and aliphatic methyl group of 2-MIM, of ZIF-67-8 at 1453 cm^{-1} and 758 cm^{-1}. According to Fig. 1B, in the pattern related to MIL101, the peaks are observed at 2θ of 5.25˚, 5.99˚, 8.57˚, 9.19˚ and 16˚, which is a confirmation of the successful synthesis of the MIL101. Figure 1B shows the XRD pattern of the synthesized MIL101@ZIF67@ZIF8 nanostructure. It can be seen that all diffraction peaks match the reported standard patterns, indicating the successful preparation of ZIF-67@ZIF8 [23].
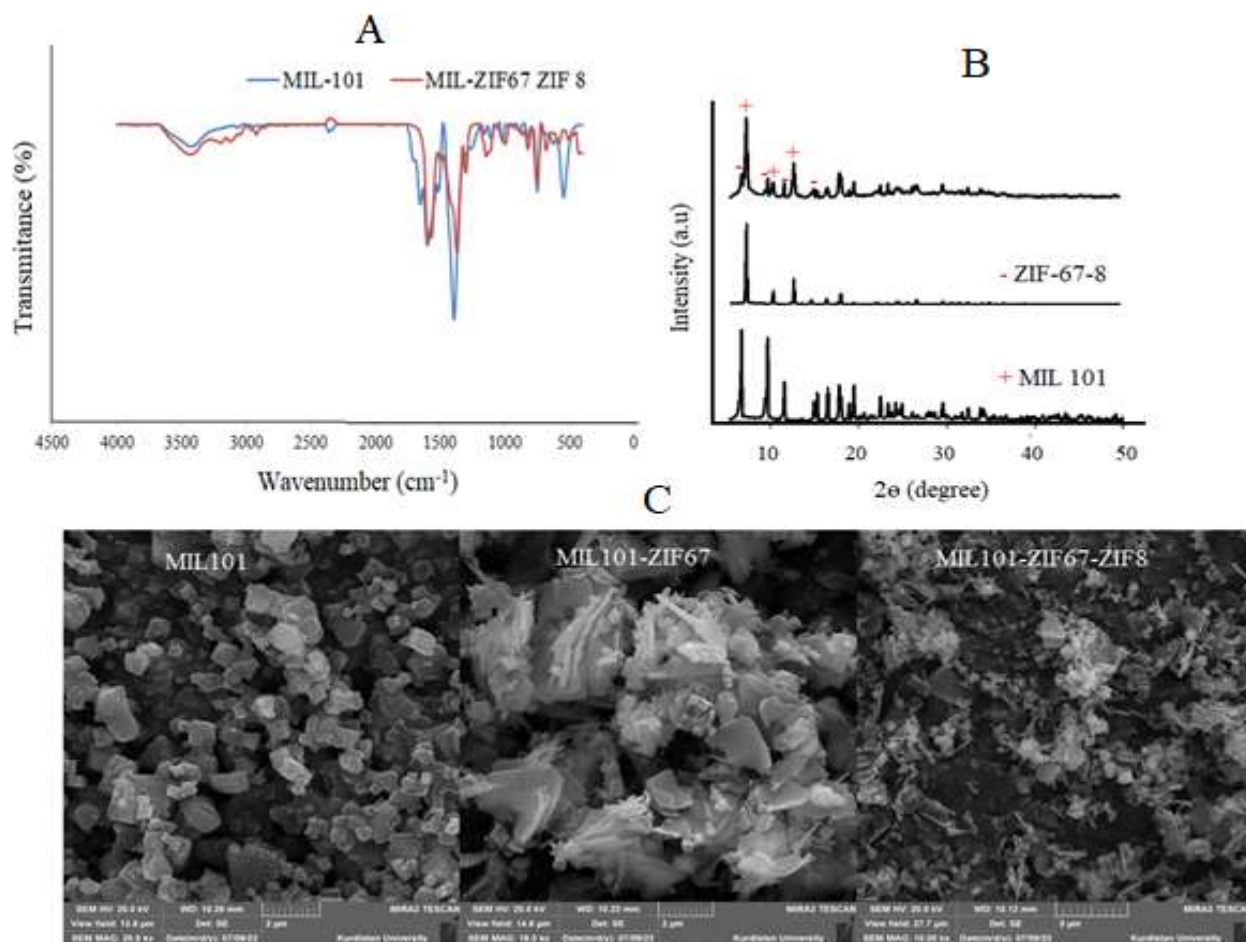
Fig. 1. FT-IR spectra of the prepared MIL101 and MIL101-ZIF67-ZIF8 (A), XRD pattern of the MIL101 and MIL101-ZIF67-ZIF8 (B), and FE-SEM image of the MIL101, MIL101-ZIF67 and MIL101-ZIF67-ZIF8 (C).

The surface morphology of MIL101 nanoparticles was done by FE-SEM. The FE-SEM image of MIL 101 clearly shows a typical octahedral structure, consistent with previous reports of MIL101. The thickness is estimated to be 20.1-64.2 nm. It is worth noting that the SEM images of the MIL101@ZIF 67@ZIF8 confirm the successful synthesis of ZIF 67 and ZIF 8 on the surface of MIL 101. Also, the results of the elemental analysis of EDS suggested the presence of C, Co, O, Zn, and Fe elements in MOF nanostructures (Fig. S1). The BET surface area of MIL-101, MIL-101@ZIF67, and MIL101@ZIF67@ZIF8 were 257, 310, and 342 m^2 g^{-1}, respectively. The total pore volume of these samples were 0.3904, 0.2918, and 0.2651 cm^3 g^{-1}. This increase in surface area and decrease in cavity volume can be

due to the fact that ZIF 67 and ZIF 8 are placed on the surface of the MIL101 and the synthesis was successful.

Optimization of Eluent Solvent Type and Concentration

It is very important to choose the right eluent solvent for the desorption of the analyte from the adsorbent surface. A good eluent should have several features. The first and most important feature of the eluent solvent is the ability to completely desorption the analyte from the sorbent surface with the smallest amount of elution solvent. Also, the eluent must be compatible with the structure and nature of the adsorbent and analyte, and not lead to the change and destruction of the adsorbent. For this purpose, the

effectiveness of different acid solutions such as HCl, H_2SO_4, HNO_3, and CH_3COOH on the desorption of Pb^{2+} and Cd^{2+} from the surface of the sorbent was investigated. Each experiment was repeated three times and the percent recovery was calculated. As can be seen in Fig. S1, the highest recovery percentage is related to the HCl solvent. It seems that when using HCl, in addition to protonation of the sorbent surface by the acid, the presence of chloride ions in very acidic environments leads to the formation of a complex between analytes and Cl^{-1}, which facilitates the process of elution of the analyte from the sorbent surface. Therefore, HCL was chosen as the elution solvent for further work.

In addition, the concentration of eluent solvent was also investigated. For this purpose, the effect of changing the concentration of HCl in the range of 0.01 to 1 M on the recovery percentage of lead and cadmium was investigated. As the results show (Fig. S2), the extraction efficiency increases with the increase of HCl concentration up to 0.5 M and remains stable after that. Therefore, it can be said that the concentration of 0.5 M of HCl is sufficient for the complete washing of analytes from the sorbent surface, and this value was chosen as the optimal concentration.

Optimization through Experimental Design

Here, in order to optimize the factors affecting the method, the experiment design strategy was used in two stages. At first, in order to identify the effective factors of the procedure, screening was done based on the Placket-Berman design, and the effective factors were identified based on the Pareto chart. In the following, these effective factors were optimized with the help of Box-Bhenken design. All information related to the experimental design process, including the Berman plate and Box-Bhenken design, is reported in the supporting data section.

Evaluation of the Method

After optimizing all the effective parameters, the calibration curve was drawn using the introduced preconcentration method. For this purpose, standard solutions were prepared from the mixture of analytes with different concentrations and extracted under the obtained optimal conditions. The calibration curve for lead and cadmium is linear with equations y = 0.0062x + 0.0154 and y = 0.0134 + 0.0457 in the concentration range 0.5-150.0 and

0.2-70.0 µg l^{-1} with correlation coefficients 0.9995 and 0.9985, respectively. The LOD of a method is defined as the lowest analyte concentration for which the response of the device is significantly different from the response of the device for the blank. The LOD was obtained from the following equation:

$$LOD = \frac{3S_b}{m}$$

where M is the slope of the calibration curve and S is the standard deviation of the blank solution. Also, the quantitative limit was obtained based on $\frac{10S_b}{m}$.

The EF values for Pb^{2+}, and Cd^{2+} ions, were 73, and 75, respectively. The %ER values for Pb^{2+}, and Cd^{2+} ions, were 91 and 93% respectively. To obtain accuracy in one day, the prepared extraction device was used to extract the desired analytes at three different concentration levels of 1, 10, and 50 µg l^{-1}. The %RSD was obtained in one day and between days, which is presented in Table 1. It is worth mentioning that the Figures of merit were also calculated for cream and urine samples and the results are listed in Table 1.

It is worth mentioning that the proposed method was compared with the works done by other researchers on cadmium and lead and the results of this comparison are presented in Table S9. From the comparison of these results, it can be found that the figures of merit of the proposed method including RSD, LOD, and LDR are comparable with other methods and the proposed method is fast and simple.

Investigating the Effect of the Presence of Interfering Ions

The selectivity of the method is one of the important parameters for determining the efficiency of the analytical method. Therefore, the effect of cations and anions that may exist in real samples along with lead and cadmium and interfere in the extraction and measurement of analytes was investigated. The interference limit is the maximum amount of foreign ions that leads to a change in the analyte signal by more than 5%. To check the interference, different proportions of disturbing ions were added to the solution containing lead and cadmium with a concentration of 50. Then, lead and cadmium were extracted and measured under optimal conditions. The results related to the interference limit of different anions and cations are reported in Table 2.

Table 1. Analytical Performance Characteristics of the Proposed Method in Water

Analyte	Linear range ($\mu g\ l^{-1}$)[a]	Coefficient of determination (r^2)				Precision (%)[b]			LOD ($\mu g\ l^{-1}$)	LOQ ($\mu g\ l^{-1}$)	EF	ER (%)
				Inter-day			Intra-day					
			1 ($\mu g\ l^{-1}$)	10 ($\mu g\ l^{-1}$)	50 ($\mu g\ l^{-1}$)	2 ($\mu g\ l^{-1}$)	10 ($\mu g\ l^{-1}$)	50 ($\mu g\ l^{-1}$)				
Water												
Pb^{2+}	0.5-150.0	0.9995	2.1	1.9	1.5	2.5	2.1	1.8	0.159	0.530	73	91
Cd^{2+}	0.2-70.0	0.9985	2.2	1.8	1.2	2.4	2.0	1.5	0.059	0.197	75	93

[a]Spiking level. [b]Relative standard deviation (RSD) (n = 3). [c]All concentrations in the cream sample are provided as $\mu g\ l^{-1}$.

Table 2. Effect of Potentially Interfering Ions on the Extraction Recovery of Target Ions (at Optimized Condition)

Ion	Added as	Ratio of interfering ions to analytes	Recovery (%)	
			Cd^{2+}	Pb^{2+}
Na^+	NaCl	5000	94	98
K^+	KCl	5000	96	99
Ca^{2+}	$CaCO_3$	5000	97	97
Ba^{2+}	$BaCl_2$	5000	95	99
Li^+	$LiNO_3$	10000	96	98
Mg^{2+}	$Mg(NO_3)_2.6H_2O$	5000	93	95
Fe^{3+}	$FeCl_3.6H_2O$	4000	93	97
Ag^+	$AgNO_3$	2000	96	99
Mn^{2+}	$Mn(NO_3)_2.6H_2O$	3000	95	98
Ni^{2+}	$Ni(NO_3)_2$	2000	94	96
F^-	NaF	5000	96	99
Cl^-	NaCl	5000	97	100
Br^-	NaBr	5000	94	97
NO^{3-}	$NaNO_3$	5000	93	99
SO_4^{2-}	Na_2SO_4	2000	92	96

Table 3. Summary of the Obtained Results from Analysis of Target Analytes in Different Real Samples by the Presented Method

		Well water						River water				
	Measured (μg l^{-1})	Added (μg l^{-1})	Found (μg l^{-1})	SR (%)	RR[a] (%)	RSD (%) n = 3	Measured (μg l^{-1})	Added (μg l^{-1})	Found (μg l^{-1})	SR (%)	RR (%)[a]	RSD (%) n = 3
Pb	ND	2.0	1.9	95	93	2.2	ND	2.0	1.9	95	96	2.8
		5.0	5.5	100	97	2.6		5.0	4.8	96	97	2.5
		10.0	10.1	101	98	3.3		10.0	10.0	100	97	2.7
Cd	ND	2.0	1.9	95	91	2.5	ND	2.0	1.8	90	92	3.1
		5.0	4.9	98	95	3.0		5.0	5.1	102	97	3.3
		10.0	9.9	99	97	3.4		10.0	9.8	98	95	3.2

		Cream						Lipstick				
	Measured (μg g^{-1})	Added (μg g^{-1})	Found (μg g^{-1})	SR (%)	RR[a] (%)	RSD (%) n = 3	Measured (μg l^{-1})	Added (μg l^{-1})	Found (μg l^{-1})	SR (%)	RR (%)[a]	RSD (%) n = 3
Pb	40.7	2.0	42.5	90	91	2.9	25.0	2.0	26.8	90	92	2.4
		5.0	45.4	94	91	2.2		5.0	30.1	102	96	2.9
		10.0	50.4	97	93	2.7		10.0	35.0	100	97	2.7
Cd	4.3	2.0	6.2	95	91	3.1	6.1	2.0	7.9	90	90	3.6
		5.0	9.3	100	97	3.5		5.0	11.0	98	95	3.2
		10.0	14.6	103	96	3.6		10.0	16.0	99	96	3.0

Real Sample Analysis

The introduced method was used to measure small amounts of lead and cadmium in various real samples such as well water, river water, cream, and lipstick. Also, certain amounts of standard solution of lead and cadmium were added to each of the real samples and after performing the extraction and measurement procedures, the recovery percentage of lead and cadmium was calculated. The results of this study are given in Table 3. As can be seen, appropriate recovery percentages were obtained in the range of 90-103, which shows that the proposed method can be successfully used to measure lead and cadmium in different real samples.

CONCLUSION

In this study, for the first time, nanoparticles MIL101@ZIF 8@ZIF67 were synthesized and used in the thin film microextraction technique to measure lead and cadmium. These nanoparticles are suitable adsorbents for lead and cadmium preconcentration due to their high absorption capacity and good stability. It should be noted that the use of this nanocomposite in the pre-concentration stage while achieving a high pre-concentration factor and a low detection limit, reduced or eliminated the effects of the texture of real samples. Therefore, the method was successfully used for sensitive, fast, simple, and low-cost analysis of lead and cadmium in real samples.

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