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Microwave-assisted synthesis of antimony oxide nanoparticles for the determination of trace cadmium in mulberry leaf tea matrices by flame atomic absorption spectrophotometry

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Abstract

In this study, a rapid, sensitive and accurate analytical method was optimized for the determination of trace levels of cadmium (Cd) by flame atomic absorption spectrophotometry (FAAS) after antimony oxide nanoparticles (AO-NPs)-based dispersive solid-phase extraction (DSPE). The AO-NPs were synthesized with a specific microwave temperature program, and they exhibited high purity and good surface morphology, making them appropriate sorbent material for the preconcentration/separation of a heavy metal. All experimental parameters affecting the extraction efficiency were optimized univariately. Under the optimum operational conditions (35 mL sample volume, 0.75 mL of pH 8.0 buffer, 15 mg of sorbent, 5 s vortex and 100 µL of 1.0 M of HNO3), the limit of detection (LOD) and the limit of quantitation (LOQ) values were determined as 0.27 and 0.89 µg L^{-1}, respectively. Thanks to the developed method, a 164.8-fold improvement in the sensitivity of the conventional FAAS system was achieved. Reusability tests showed that the AO-NPs can be employed 5 times. The feasibility of the method was confirmed by recovery tests with mulberry leaf tea matrices, and good recovery results between 77.6 and 115.8% were obtained using the matrix matching calibration method.

Keywords Cadmium, Antimony oxide, Flame atomic absorption spectrophotometry, Mulberry leaf tea, Microwave synthesis

Introduction

Heavy metals are nonbiodegradable toxic pollutants that tend to accumulate in living organisms and are known to threaten human health and ecology by spreading through the food chain (Verma et al. 2023). The widespread use of heavy metals for native, industrial, and agricultural purposes increases the possibility of human exposure to these metals (Divrikli et al. 2006; Jayamurali et al. 2021). Metallic ions, which exist in various species in the environment, differ in physical and chemical features and their toxicity to living organism (Ali et al. 2005; Ali and Gupta 2006). Accordingly, reactive oxygen species (ROS) that generate oxidative stress have been reported to be hazardous species for cells. Moreover, the presence of

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metal toxins results in the depletion of the antioxidant system of the body (Jayamurali et al. 2021).

Cadmium (Cd), which is known to be a potentially toxic element, occurs in the environment because of natural phenomena and human actions. Natural phenomena such as volcanic activity, rock and soil erosion, and forest fires contribute to elevated Cd levels in the natural environment including the atmosphere, soil, and water. On the other hand, various human activities such as burning of fossil fuels, refining and smelting of copper/nickel, and the use of phosphate fertilizers lead to the increase in Cd concentrations in the environment (Genchi et al. 2020). Cd is widely employed in various industrial products/processes including pigments, neutron absorbers in nuclear power plants, anticorrosive agents, stabilizers in plastic products and the production of cadmium–nickel batteries (Godt et al. 2006). Humans are exposed to this element through the respiratory system and gastrointestinal tract by inhalation of polluted air and cigarette smoke, and consumption of contaminated food (Satarug 2018). Cadmium and its compounds are recognized as carcinogens by both the International Agency for Research on Cancer and the National Toxicology Program due to its potential to damage the bones, kidneys, and lungs (Godt et al. 2006; Huff et al. 2007). Therefore, it is important to determine Cd in environmental samples at low concentrations with high accuracy.

A variety of analytical techniques have been applied over the past years to quantify Cd levels in different samples, and these techniques include UV–Vis spectrophotometry (Wen et al. 2011), inductively coupled plasma-mass spectrometry (ICP-MS) (Hutton et al. 2004), inductively coupled plasma-optical emission spectrometry (ICP-OES) (Massadeh et al. 2010), hydride generation atomic fluorescence spectrometry (HGAFS) (Luo et al. 2017), graphite furnace atomic absorption spectrometry (GFAAS) (Korn et al. 2010), electrothermal atomic absorption spectrometry (ETAAS) (Pyrzynska 2007), and flame atomic absorption spectrometry (FAAS) (Meira and de Souza Dias 2017). Among these analytical techniques, FAAS stands out because of its economical operation, ease of use and quick response. Moreover, in this instrument, metal ions can be easily atomized in an air-acetylene flame and are not prone to interference (Ferreira et al. 2007). However, performing the direct determination of elements using FAAS can be challenging due to its limited detection capability for most analytes, which are present in samples at low concentrations. Furthermore, low nebulization efficiency and interferences due to complex sample matrix are other limitations of FAAS systems. Consequently, in order to overcome these disadvantages, it is often necessary to perform an appropriate sample preparation procedure prior to determination, followed by separation of the target analyte(s) from the matrix (Alves et al. 2010; Fernández et al. 2019; Liang and Han 2006).

Several sample preparation methods such as ultrasound-assisted surfactant-enhanced emulsification microextraction (UASEME) (Yao et al. 2018), dispersive liquid–liquid microextraction (DLLME) (Rojas et al. 2011), cloud point extraction (CPE) (Golbedaghi et al. 2012), solid-phase extraction (SPE) (Tunçeli et al. 2019), solidified floating organic drop microextraction (SFODME) (Alahabadi et al. 2017), and dispersive solid-phase extraction (DSPE) (Islam et al. 2019) have been developed for effective separation of analyte(s) from different matrices to improve the precision of Cd determination at trace levels. The DSPE method is based on the dispersion of the sorbent compound in the sample solution and adsorption of the analyte(s) on sorbent surface. The DSPE method minimizes the drawbacks of the traditional SPE method such as low extraction efficiency, long equilibration time, and high solvent consumption (Yang et al. 2020). Recently, numerous sorbent materials with different chemical/physical properties have been reported in literature for the separation of analyte(s) from sample matrices. Nanoparticles (NPs) are interesting materials produced from organic/inorganic compounds with high ability to adsorb different pollutants due to their nanoscale size, high porosity, and binding capacity of active sites on the surface (Ali et al. 2022, 2019; Georgin et al. 2024). In terms of their physical properties, nanoparticles (NPs) are innovative types of materials having dimension(s) smaller than 100 nm (Ali et al. 2023; Khan et al. 2019). NPs have great potential as adsorbents and catalysts for many research areas due to their chemical reactivity and large adsorption surface in addition to other properties. Thus, target analytes can easily adsorb and interact with other ions to stabilize the ion charge (Yu et al. 2021).

Antimony oxides are generally known for their existence in various phases (Sb_2O_3, Sb_2O_4, Sb_2O_5, Sb_6O_{13}) and polymorphic forms. These different phases have attracted considerable interest in different fields including optics, catalysts, medicine, and semiconductors (Liu et al. 2014). The α-Sb_2O_4 form has been widely used in catalysis (Adimule et al. 2021).

There are various methods reported in literature for the synthesis of a variety of nanoparticles and these include solvothermal, thermal decomposition, coprecipitation, combustion, chemical vapor deposition, microemulsion, laser pyrolysis, carbon arc, sonochemical, and microwave-assisted techniques (Majidi et al. 2016). Among these, microwave-assisted nanoparticle synthesis is an innovative method to heat reaction solutions homogeneously and rapidly. In this system, interactions between molecules are accelerated by microwave radiation equipped with both magnetic and electrical components (Ding et al. 2003).

The leaves of the mulberry tree are rarely consumed as food, but they are mainly used to make mulberry leaf tea mostly in Asian countries for its health benefits (Makchuay et al. 2023). Because of its high content of active phenolic compounds, such as astragalin, kaempferol, isoquercitrin, quercetin, rutin, and chlorogenic acid, mulberry has been regarded as antioxidative, antihyperglycemic, anti-inflammatory, glucose lowering, cholesterol lowering, and cardioprotective (Ruengdech et al. 2019). Mulberry trees are prone to heavy metal pollution due to their location in contaminated soils, the use of synthetic fertilizers, and irrigation water containing heavy metals. The subsequent consumption of crops harvested from such trees leads to the transfer of heavy metals into the consumer (Yang et al. 2021). Therefore, monitoring heavy metal levels in mulberry tree products is vital for protecting human health and general well-being.

Determination of trace levels of cadmium using AO-NPs is a novel approach suggested by in this study. Trace levels of cadmium in mulberry leaf tea extract were determined under the optimum conditions of the AO-NP-DSPE-FAAS method, which were determined after performing univariate optimization of each parameter. This work represents the first study in the literature on the usage of AO nanoparticles as sorbent materials for the separation/preconcentration of a heavy metal from a sample matrix. There is study in the literature where this nanoparticle is used for the determination of cadmium.

Materials and methods

Reagents and chemicals

Analytical grade chemicals and reagents were used throughout the study. Working standard solutions of cadmium at different concentrations were prepared from a 1000 mg L^{-1} stock solution purchased from Merck (Germany). The pure water (18.2 MΩ.cm resistance) used for processes including sample/standard dilution and washing was obtained from an Elga Pure Flex 3 Ultrapure Water System (UK). Buffer solutions were prepared in the pH range of 5.0 and 10 using Tris and potassium hydrogen phthalate bought from Merck, and sodium tetraborate decahydrate purchased from Sigma-Aldrich (Germany). In the synthesis of AO nanoparticles, antimony trichloride and urea were purchased from Sigma Aldrich and Tekkim Kimya (Türkiye), respectively. For pH adjustment, sodium hydroxide and hydrochloric acid were purchased from Isolab (Türkiye) and prepared in dilute concentrations. Acetylene gas used as fuel to generate the flame required for atomization was purchased from a local supplier in İstanbul, Türkiye. The mulberry leaves used for the recovery studies were collected from two different trees in İstanbul, Türkiye.

Instrumentation

Cadmium levels were monitored with an ATI Unicam 929 AA model flame atomic absorption spectrophotometer (FAAS) with a deuterium lamp (D_2) for background corrections. A hollow cathode lamp with an operating current of 15 mA and a wavelength of 228.8 nm was used as a continuous light source. A Velp, ZX3 model vortex mixer and Heidolph brand mechanical stirrer were used for the mixing processes. Mettler Toledo pH meter was used for pH adjustment during the nanoparticle synthesis procedure and buffer solution preparation. Nanoparticle synthesis was conducted in the Milestone Microwave Digestion System. A biobased BKC-TL5II centrifuge was used for phase separation. The nanoparticles were held in an Heraeus oven for the removal of possible moisture.

Synthesis of AO nanoparticles by microwave process

Based on a work reported in the literature, a synthesis procedure was developed to synthesize the AO nanoparticles (Adimule et al. 2021). First, 0.015 mol $SbCl_3$ and 0.02 mol urea ($CO(NH_2)_2$) were dissolved in ultrapure water and the solution was stirred on a mechanical stirrer for 30 min. Then, 1.0 M of NaOH solution was added dropwise with continuous stirring until the pH of the solution reached 10.5. In the next step, the final solution was transferred into a Teflon container of the microwave system. The temperature program used for the synthesis of the AO nanoparticles was as follows: 5.0 min temperature ramp to 100 °C (5.0 min hold time) and 5.0 min ramp to 150 °C (25 min hold time), followed by a cooling time of 10 min as the final step. After the synthesis was completed, the reaction solution was transferred into a 50 mL conical tube and centrifuged at 3000 rpm for four minutes to separate the nanoparticles from solution. The nanoparticles were washed 3 times with pure water and 2 times with acetone for the removal of possible impurities. The washed AO-NPs were dried in an oven at 50 °C for 24 h. Finally, calcination of the nanoparticles was performed at 550 °C for 2 h. The prepared nanoparticles were characterized using scanning electron microscope (SEM) and X-ray diffraction (XRD) techniques (Fig. 1). The surface morphology of the NPs was examined by SEM, which showed that the synthesized sorbent is compatible with the definition of nanoparticles by IUPAC (Vert et al. 2012). The XRD pattern verified the crystal structure of the Sb_2O_4 NPs by scanning in the angular range 10–90° of 2θ with a step size of 0.008° at 45.0 kV and 40.0 mA of radiation generation setting. The pattern seen at $2\theta = 28.37°$ indicates an orthorhombic Sb_2O_4 phase (Balamurugan et al. 2023), and the crystal

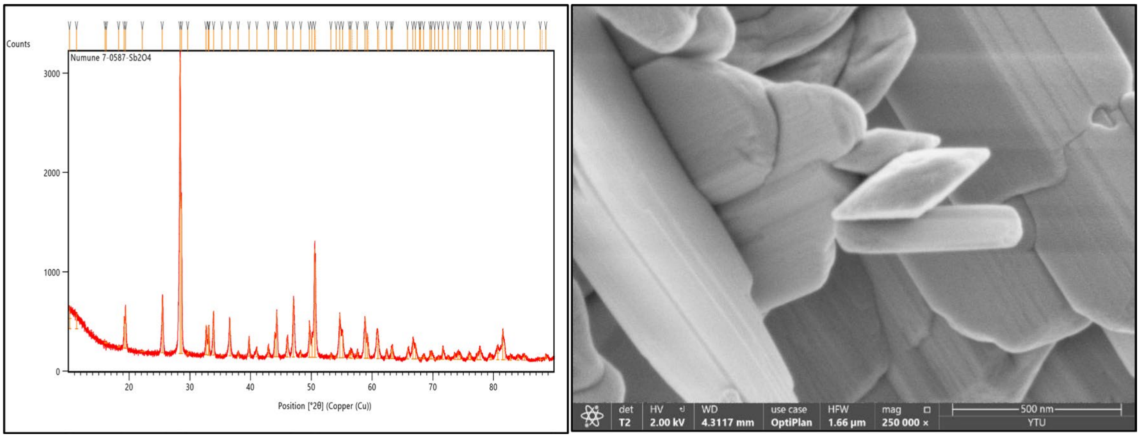

Fig. 1 XRD (left) and SEM (right) images of produced AO-NPs

structure of the NPs was confirmed by other XRD patterns by comparing with the literature findings including intensity and 2θ degree scales (Adimule et al. 2021; Tadjarodi and Karimpour 2014; Yadav et al. 2010).

Samples

The mulberry leaves were plucked and allowed to dry naturally in a sterile environment until they reached the recommended dryness for consumption. Then, 2.5 g of each mulberry leaf sample was weighed, and 200 mL of distilled water was added and allowed to infuse for 10 min. The solution was then filtered through a filter paper to remove the pulp, and the final volume was diluted to 2000 mL with distilled water. The final solution was used as a sample solution to prepare the concentrations determined for the recovery tests.

Procedure of AO-NP-DSPE-FAAS method

Approximately 15 mg of AO nanoparticles were weighed into falcon tubes. 35 mL of sample and 0.75 mL of pH 8.0 buffer solution were added to the nanoparticles and then mixed by vortex for 5 s. To facilitate phase separation, centrifugation was performed at 3000 rpm for an optimum period. The supernatant was decanted to allow access to the analyte adsorbed nanoparticles at the bottom of the sample tubes. For the elution of cadmium ions from the surface of the nanoparticles, 100 µL of 1.0 M HNO_3 was used. In the final step, the extracted phase was analyzed by FAAS.

Results and discussion

In the optimization studies, the acidity of solution/buffer volume, sample volume, sorbent mass, eluent concentration/volume, and mixing time parameters, which are critical parameters in increasing the extraction efficiency and detection power of the FAAS system, were optimized with a univariate optimization approach in three replicate analyses.

Effect of pH and buffer volume

The acidity of a sample solution is crucial for the adsorption of target analytes onto the surface of a sorbent material. As the pH of the medium decreases, hydronium ions hinder the transfer of analyte ions to the sorbent surface, resulting in lower extraction efficiency (Al-Senani and Al-Fawzan 2018). For this reason, the pH of the standard solutions was adjusted by adding buffer solutions varying between 5.0 and 10 and extraction was performed to evaluate the effect of pH on adsorption efficiency under equal experimental conditions. The results showed that the mean absorbance values increased linearly from pH 5.0 to pH 8.0. Here, the increase in the mean absorbance values toward the alkaline region is attributed to the increased adsorption of Cd ions on the surface of the AO nanoparticles. On the other hand, the mean absorbance values between pH 8.0 and 10 were very close to each other. Since it is reported in the literature that dissolved Cd starts to precipitate in the hydroxyl form after pH 8.0, the optimum sample solution pH was chosen as 8.0 (Salmani et al. 2013). Considering the literature findings, Revya-Ramos et al. reported the highest adsorption efficiency at pH 8.0 for Cd removal from aqueous samples using activated carbon obtained from coconut shells (Leyva-Ramos et al. 1997). Wang et al. proposed a method for Cd adsorption from aqueous solutions at pH 8.0 using bamboo charcoal as a sorbent (Wang et al. 2010). As reported in both reports, as the pH of the medium increases, the number of negatively charged sites on the surface of the adsorbents increases. Therefore, the adsorption efficiency of cationic Cd (II) ions may have increased due to electrostatic attraction.

Subsequently, extractions were carried out by adding different volumes in the range of 0.50–2.0 mL to aqueous solutions to determine the optimum buffer volume. The signals increased from 0.50 to 0.75 mL, whereas close absorbance values were recorded for the other volumes. Since the mean absorbance values were very close to each other, 0.75 mL was selected as the optimum buffer volume.

Effect of sample volume

One of the parameters that plays a key role in increasing absorbance efficiency is sample volume. Theoretically, as the analyte ion concentration in the sample solution increases with increasing sample volume, the ratio of initial volume to final volume is directly related to the enhancement of detection power. Therefore, optimizing the sample volume is important for high extraction efficiency (Naeemullah et al. 2012). In this step, different sample volumes between 20 and 40 mL in 5.0 mL increments were transferred to a 50 mL falcon tube and extraction process was performed under the same experimental conditions. The maximum sample volume was limited to 40 mL for efficient mixing, considering the volume and shape of the utilized falcon tubes. As shown in Fig. 2, the mean absorbance values increased from 20 to 35 mL and very close measurement results were obtained for 35 and 40 mL. These close results between 35 and 40 mL support the assertion that saturation occurred on the surface of AO-NPs (Maranata et al. 2021). Hence, 35 mL was determined as the optimum volume of the sample in further experiments.

Effect of sorbent amount

In extraction studies, the amount of sorbent is one of the parameters that directly impacts extraction efficiency. For a certain sample volume, excess amounts of a sorbent will provide more surface area, but increased solvent consumption during the elution process will take the method away from the environmentally friendly/greenness concept. Conversely, the sorbent surface area required for an efficient extraction is limited due to the low amount of sorbent consumption, resulting in low extraction outputs for target analyte(s) (Çelik et al. 2018). In addition, excessive use of sorbent results in increased cost per unit of analysis and wastage of sorbent (Altunay et al. 2022). Hence, AO-NPs between 15 and 50 mg were added to standard solutions to determine the optimum sorbent amount and extraction processes were carried out under equal experimental conditions. The results presented in Fig. 3 show that the mean absorbance values were close to each other. Subsequently, the effects of 15 mg and lower sorbent amounts (7.5, 10, and 12.5 mg) on the extraction efficiency were investigated. The mean absorbance values increased from 7.5 to 15 mg, and the highest signals were recorded for 15 mg. Therefore, 15 mg was selected as the optimum sorbent amount.

Effect of eluent concentration/volume

The desorption or elution process is critical for the release of the analyte(s) transferred to the sorbent surface from the bulk sample, and the evaluation of adsorption efficiency. In this process, eluent type, eluent volume, and eluent concentration are three important parameters that play a key role in the desorption of analytes and the achievement of a high enrichment factor (Shokrolahi et al. 2010). Since acid solutions generate protonation in the adsorption zone of the sorbent material, acid solutions are often used to release metal ions in the elution process. Among these solvents, nitric acid is a protic solvent that provides high efficiency for the desorption of metal ions from the sorbent surface and does not cause interference in spectroscopic measurements (Timur et al.

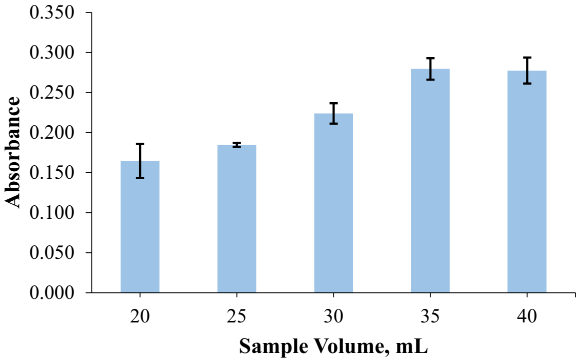

Fig. 2 Influence of sample volume on extraction outputs (0.75 mL of pH 8.0, 50 mg AO-NPs, 30 s vortexing, 0.25 mL of 7.0 M HNO_3), (*n* = 3)

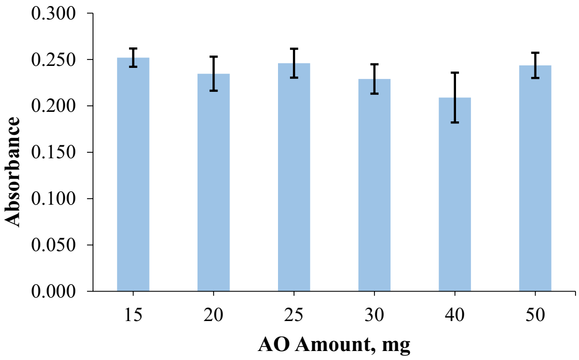

Fig. 3 Influence of sorbent amount on extraction outputs (35 mL of 25 µg L^{-1} Cd standard solution, 0.75 mL of pH 8.0, 30 s vortexing, 0.25 mL of 7.0 M HNO_3), (*n* = 3)

2009). In this step, different concentrations of HNO_3 in the range of 0.25–14.4 M were employed under equal experimental conditions to investigate the desorption ability of HNO_3 for cadmium ions from the surface of AO-NPs. According to the results given in Fig. 4, the mean absorbance values increased from 0.25 to 1.0 mL and reached the highest value at 1.0 mL. After 1.0 M, the mean absorbance values decreased as the HNO_3 concentration increased. Rocha et al. reported that a decrease in nebulizer efficiency may occur as viscosity and density increase with increasing acid concentration (Rocha and Nòbrega 1996). 0.25 and 0.50 M of HNO_3 did not have sufficient acidity to desorb cadmium ions from the sorbent surface. Therefore, 1.0 M was determined as the optimum HNO_3 concentration.

Next, varying volumes (100, 125, 150, and 200 µL) of 1.0 M of HNO_3 were tested to evaluate the elution capacity of cadmium ions. The results depicted in Fig. 5 demonstrated that the highest signal-to-noise ratio was obtained for 100 µL, which gradually decreased with increasing volumes. Here, one of the main reasons for the decrease in responses is the dilution of the analyte with increasing volume. In order to investigate the elution capacity at lower volumes, elution was attempted by adding 75 µL of 1.0 M of HNO_3, but sufficient extraction phase for feeding into the system during phase separation could not be achieved. Therefore, 100 µL was determined as the optimum eluent volume.

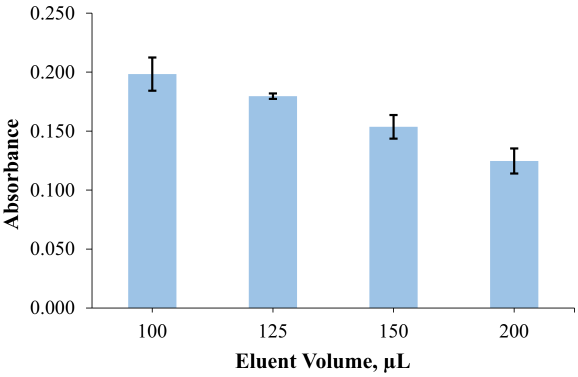

Fig. 5 Influence of eluent volume on extraction outputs (35 mL of 10 µg L^{-1} Cd standard solution, 0.75 mL of pH 8.0, 15 mg AO-NPs, 30 s vortexing), ($n=3$)

Effect of vortexing period

Mixing process, which accelerates the transfer of analytes to the sorbent surface by increasing the homogeneous distribution of the sorbent material in solution, is of high importance for an effective extraction process. In addition, achieving high extraction outputs in a short time makes the proposed method more attractive in terms of applicability and preferability (Sanmartín et al. 2022). The extraction efficiency was evaluated at 5.0, 10, 20, and 30 s of vortex, which is one of the mechanical forces frequently used for sample agitation. In the results presented in Fig. 6, the highest mean absorbance value was recorded at 5.0 s. The interaction between Cd ions and AO started to decrease by about 10% for 10 and 20 s and by about 20% for 30 s. The results were compared with the same experimental condition performed without mixing to demonstrate the effect of mixing on the extraction efficiency. The mean absorbance values obtained for 5.0 s were about 2.5 times higher than the mean absorbance values obtained without mixing. Therefore, 5.0 s was chosen as the optimum mixing period by vortex, which enhances the rapidness of the analytical method.

Analytical performance of the method

System analytical performance values of the developed AO-NP-DSPE-FAAS method were evaluated under the optimum conditions given in Table 1.

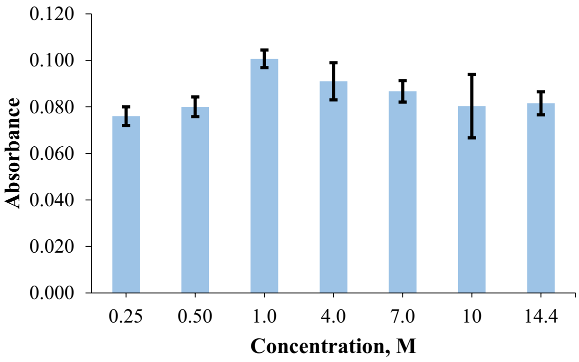

Fig. 4 Influence of eluent concentration on extraction outputs (35 mL of 10 µg L^{-1} Cd standard solution, 0.75 mL of pH 8.0, 15 mg AO-NPs, 30 s vortexing, 0.25 mL of eluent), ($n=3$)

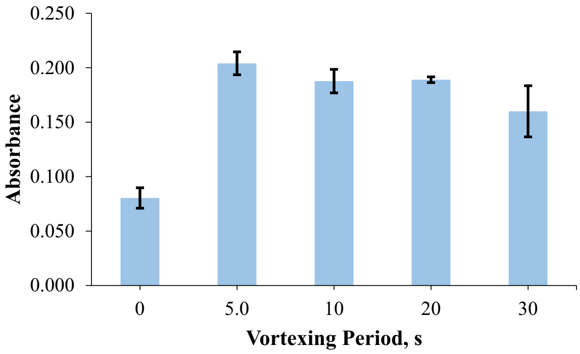

Fig. 6 Influence of vortexing period on extraction outputs (35 mL of 10 µg L^{-1} Cd standard solution, 0.75 mL of pH 8.0, 15 mg AO-NPs, 100 µL of 1.0 M of HNO_3), ($n=3$)

Table 1 Optimum operational conditions for the AO-NP-DSPE-FAAS method in the adsorption/preconcentration of cadmium

Experimental parameters	Optimum condition
Sample volume	35 mL
pH/Buffer volume	pH 8.0/0.75 mL
AO-NPS AMOUNT	15 mg
Vortexing period	5.0 s
Eluent type/concentration/volume	1.0 M of HNO_3/100 µL

The standard deviation value calculated from the lowest concentration in the calibration plot was divided by the slope of the plot and multiplied by three and ten to calculate the limit of detection (LOD) and limit of quantitation (LOQ) values, respectively. Correspondingly, the LOD and LOQ values were calculated as 0.27 and 0.89 µg L^{-1}, respectively. The percent relative standard deviation (%RSD) of the method with a linear range of 0.50–20 µg L^{-1} was 14.5% for ten replicates of the lowest concentration. The linear calibration plot equations (standard concentrations in µg L^{-1}) for the FAAS and AO-NP-DSPE-FAAS systems were $y = 0.0001098x + 0.0059$ and $y = 0.0181x + 0.0015$, respectively, and regression coefficients were greater than or equal to 0.9983 for each system. Based on the slope of the linear calibration equation of the developed AO-NP-DSPE-FAAS method, a 164.8-fold improvement in conventional FAAS sensitivity was achieved. A detailed summary of the figures of merit is presented in Table 2.

Reusability test

The reusability of the sorbent used in an adsorption process is a very significant advantage for the reduction of chemicals, energy, cost, time, and labor when a new adsorbent is required. Moreover, since sorbent waste at the end of the process is a concern for the environment, reusable sorbents are of great interest to minimize the rate of disposal into the environment. In order to investigate the reusability of the nanomaterial, the used NPs were washed with the elution solution after the first optimization and then dried after removing possible impurities with acetone and water as mentioned in the last step of the synthesis procedure. Extraction results of these NPs and the original NPs were compared under similar conditions, and the mean absorbance values were found to be very close to each other. Further reusable extractions were performed, and it was verified that the AO nanoparticles can be used fully efficiently for five cycles.

Evaluation of other reports on cadmium preconcentration in the literature

The analytical performance of other reported literature findings for the separation/deposition of cadmium was compared with the AO nanoparticle-based DSPE-FAAS method of this study (Table 3).

Melo et al. 2000 achieved an LOD of 1.2 µg L^{-1} with a 108-fold improvement in the determination power of the FAAS system by using 100 mg of Amberlite XAD-2 resin for the preconcentration of Cd (Melo et al. 2000). Jesus et al. 2012 obtained an LOD of 0.20 mg kg^{-1} by direct solid sampling in a GFAAS system for the direct determination of Cd in crude oil matrices (de Jesus et al. 2012). El Sheikh et al. 2019 recorded an LOD value of 1.8 µg L^{-1} after preconcentration of cadmium in tap water and well water samples approximately 10 times using 50 mg of magnetite and magnetic carbon nanotube nanoparticles in the method they developed in FAAS system (El-Sheikh et al. 2019). Suleiman et al. 2008 reported the determination of low levels of cadmium in biological samples by developing a microcolumn-assisted flow injection method. The 20 mg of microcolumn was loaded as sorbent. Here, the LOD and improvement of detector power were 0.30 µg L^{-1} and 20, respectively (Suleiman et al. 2008). Khan et al. developed a modified graphene oxide-assisted extraction method for FAAS in 2016 for the determination of Cd ions at low levels in natural water and food matrices. A 40-fold improvement in sensitivity was achieved, and the LOD was reported as 0.37 µg L^{-1} employing 100 mg of sorbent (Khan et al. 2016). Yılmaz et al. 2023 developed a vortex-assisted dispersive solid-phase microextraction method to separate Cd ions from water, soft drink, and food samples for determination in an FAAS system. Here, 250 mg of magnetic polystyrene-b-poly dimethyl siloxane hydrophobic block copolymer was utilized as sorbent material, yielding enhancement in sensitivity and the LOD values of 50 and 1.7 µg L^{-1}, respectively (Yılmaz et al. 2023). In de S. Dias et al. 2019 proposed a magnetic solid-phase microextraction

Table 2 System analytical performances of the different FAAS systems

Method	LOD, µg L^{-1}	LOQ, µg L^{-1}	R^2	Linear Range, µg L^{-1}	Equation	Enhancement in sensitivity
FAAS	26.9	89.5	0.9983	100–5000	$y = 0.0001098x + 0.0059$	–
AO-NP-DSPE-FAAS	0.27	0.89	0.9988	0.50–20	$y = 0.0181x + 0.0015$	164.8

Table 3 A comparison between the developed method and the other reports

Method	LOD, μg L^{-1}	LOQ, μg L^{-1}	R^2	Linear Range, μg L^{-1}	pH	Sorbent Mass, mg	PF*	Related study
Online SPE-FAAS[a]	1.2	–	0.9997	0–200	8.24	100	108 (Enrichment Factor)	Melo et al., (2000)
DS-GFAAS[b]	0.20 μg kg^{-1}	0.70 μg kg^{-1}	0.9992	1.0–5.0	–	–	–	de Jesus et al., (2012)
MCNT-MSPE-FAAS[c]	1.8	6.1	–	6.1–300	8.2	50	10	El-Sheikh et al., (2019)
μC-FI-ICP-OES[d]	0.30	–	0.9997	20–3000	7.0	20	20 (Enrichment Factor)	Suleiman et al., (2008)
MGO-DVB-VA-MSPE-FAAS[e]	0.37	–	0.9990	–	6.0	100	40	Khan et al., (2016)
VA-DSPME-FAAS[f]	1.7	–	0.9918	5.1–100	6.0	250	50	Yılmaz et al., (2023)
$CoFe_2O_4$-NP-SPME-FAAS[g]	0.24	0.80	0.9980	–	10	25	31.5	de S. Dias et al., (2019)
AO-NP-DSPE-FAAS[g]	0.27	0.89	0.9988	0.50–20	8.0	15	164.8	This study

[a] Online enrichment based on solid-phase extraction–flame atomic absorption spectrometry

[b] Direct solid sampling graphite furnace atomic absorption spectrometry

[c] Magnetic carbon nanotube-based magnetic solid-phase microextraction–flame atomic absorption spectrometry

[d] Microcolumn flow injection-inductively coupled plasma-optical emission spectrometry

[e] Magnetic allylamine-modified graphene oxide-poly(vinyl acetate-co-divinylbenzene) nanocomposite-based vortex-assisted magnetic solid-phase extraction flame atomic absorption spectrometry

[f] Vortex-assisted dispersive solid-phase microextraction–flame atomic absorption spectrometry

[g] Cobalt ferrite nanoparticles-based solid-phase microextraction–flame atomic absorption spectrometry

[h] Antimony oxide nanoparticles-based dispersive solid-phase extraction–flame atomic absorption spectrometry

[*] Preconcentration factor

method for the determination of Cd in oyster and water samples using $CoFe_2O_4$ nanoparticles. An LOD value of 0.24 μg L^{-1} and a 31.5-fold enhancement factor were obtained utilizing 25 mg of sorbent (de S. Dias et al. 2019). Considering the experimental outputs of these reports, the analytical performance of the proposed analytical strategy for the determination of Cd is close to the analytical performance values of costly and sensitive techniques such as ICP-OES and GFAAS, indicating that it can be a good alternative to these systems. Furthermore, the proposed AO-NP-DSPE-FAAS method offers a competitive and preferable alternative for the separation/preconcentration of Cd compared to other methods with the advantages of very low sorbent amount requirement, lower volume, and lower concentration of solvent consumption and very short extraction time. Moreover, the reusability of Sb_2O_4 nanoparticles for 5 cycles and their first usage in an preconcentration/extraction study offer novelty with low-cost compared to other methods.

Recovery experiments

The best way to investigate the applicability of a developed method is to apply it to a real sample matrix and then evaluate it based on recovery percentages. Accordingly, samples of mulberry leaf tea collected from different trees were used to validate the method. Mulberry leaf tea, which is consumed by humans due to the restorative effects of its phenolic components on metabolic functions, was selected as the real sample for the recovery experiments due to the possibility of exposure to cadmium through soil in the area where the mulberry tree is located/grown. In preliminary experiments, sample analyses were performed on tea samples to investigate the presence of cadmium, but no analytical signal was recorded in the tea samples. Subsequently, the tea samples were spiked to final concentrations of 5.0, 7.5, 10, and 12.5 μg L^{-1}, and subjected to the optimal experimental conditions presented in Table 1. The calculated percent recovery results are summarized in Table 4.

The recovery values calculated using the external calibration method were in the range of 51.5–61.6%, and this suggested that compounds in the mulberry leaf tea had a suppressive effect on the cadmium signal. One of the preferred methods to minimize matrix effects in the determination of target analytes with high accuracy is the matrix matching calibration method. For this purpose, the measurement results were reassessed by matrix matching calibration and good recoveries between 77.6 and 115.8% were calculated.

Conclusion

An efficient, rapid, and environmentally friendly analytical strategy (AO-NP-DSPE-FAAS) was developed for the trace determination of cadmium in mulberry leaf tea matrices. The method involved the use of a microwave system to synthesize homogenous AO-NPs, employed as sorbents for the extraction process. This study is the first to use AO-NPs as sorbent in the

Table 4 Recovery results of mulberry leaf tea matrices using external and matrix matching calibration ($n=4$)

	Final concentration, μg L^{-1}	EC Recovery ± SD, %	MMC Recovery ± SD, %
TEA SAMPLE 1	5.0	52.4 ± 5.6	77.6 ± 11.3
	7.5	57.2 ± 1.3	96.8 ± 2.7
	10	51.5 ± 3.8	89.9 ± 7.7
	12.5	53.1 ± 5.2	96.1 ± 10.5
TEA SAMPLE 2	5.0	60.0 ± 6.0	110.6 ± 11.7
	7.5	61.6 ± 6.2	115.8 ± 12.0
	10	57.8 ± 3.7	109.5 ± 7.3
	12.5	53.9 ± 0.4	102.5 ± 0.7

SD: Standard Deviation, EC: External Calibration, MMC: Matrix Matching Calibration

determination of heavy metals. Under the optimum experimental conditions, the LOD value was found to be 0.27 μg L^{-1}, providing a 164.8-fold improvement in the sensitivity of the conventional FAAS method. When the outputs of the method are compared with the literature findings, the AO-NP-DSPE-FAAS method can compete with expensive and complex devices for cadmium determination due to its low sorbent amount and high preconcentration factors. The fact that AO-NPs can be used 5 times increases the preferability of this sorbent for different experimental applications. The feasibility of the method was evaluated by spiking-based recovery studies on matrices of mulberry leaf tea, one of the herbal tea species, and good recovery results between 77.6 and 115.8% were obtained by the matrix matching calibration method. These results show that the proposed method can be applied to mulberry leaf tea and similar matrices with high accuracy and precision.

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Author contributions

Merve Nur İNCE contributed to data curation, formal analysis, methodology, validation, visualization, and writing—original draft. Hakan SERBEST was involved in data curation, formal analysis, investigation, methodology, validation, visualization, and writing—original draft. Sezgin Bakırdere contributed to conceptualization, data curation, investigation, methodology, supervision, validation, and writing—review and editing.

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Availability of data and materials

Data will be made available with reasonable request.

Declarations

Ethical approval and consent to participate

This article does not contain any studies with human participants or animals performed by any of the authors.

Competing interests

The authors declare that they have no conflicts of interest.

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