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Waste toner particles based dispersive solid phase extraction for the trace determination of cadmium in cinnamon tea samples by flame atomic absorption spectrometry

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ABSTRACT

This study presents a dispersive solid phase extraction (DSPE) protocol for the sensitive and accurate determination of trace levels of cadmium ions (Cd^{2+}) by flame atomic absorption spectrometry (FAAS). Toner particles obtained from used printer cartridges were used as sorbent material for preconcentration of Cd^{2+} ions. Optimization studies were performed univariately for the achievement of the highest extraction efficiency and the limit of detection (LOD) was found to be 0.55 μg L^{-1} under optimal operating conditions. By comparing the slopes of the linear plot equations for FAAS and waste toner-based DSPE-FAAS systems, the increase in sensitivity was calculated to be 40.9 times. The feasibility of the developed method was evaluated by spiking recovery studies on cinnamon tea samples and good recovery results between 90.4 % and 119.0 % determined by the matrix matching calibration strategy showed that the method is applicable to cinnamon tea and similar matrices.

1. Introduction

As a result of an increasing population and expanding industry worldwide, there has been a global growth in the demand for technological tools. The electronic devices such as information technology/telecommunication devices and household electrical appliances are integral to everyday life, but they also generate significant amounts of e-waste (Choudhary et al., 2024). Ink cartridges from printers remain a major source of electronic waste, even in today's current trend of digitalization. Cartridge-based printing devices containing toner powder are the main printing materials commonly employed in office and printing applications. Considering that printing and copying activities are widespread around the world, the amount of printer cartridge waste generated globally is in the millions of tons (Babar et al., 2019). Typically, the composition of toner in cartridges is composed of iron, carbon, silicon, and a binder polymer, and contains different compounds depending on the specific formulations of the printer manufacturers (Choudhary et al., 2024). Based on the toner content, the transformation of waste materials into value-added products creates the opportunity for several innovative studies based on the principle of green chemistry (Parthasarathy, 2021).

On a global scale, heavy metal pollution poses a significant problem for environmental resources. Even at low concentrations, heavy metals are major contaminants of food sources essential for living organisms on the earth. Heavy metals have the potential to penetrate the vital cellular components of human, animal and plant tissues and, after transfer to the human body, can bioaccumulate and cause organ dysfunctions (Kwon et al., 2024). Cadmium (Cd), one of the Group 1 carcinogenic elements, can remain in environmental sources for long periods of time without degradation, and can bioaccumulate by penetrating water and food sources for human consumption and reaching metabolism through the food chain. Kidney, bone and liver diseases, different types of cancer and

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DNA damage are some of the diseases caused by accumulation of low concentrations of Cd (Dong et al., 2024). The maximum allowable levels for Cd ions in drinking water have been set by the World Health Organization (WHO) and US Environmental Protection Agency (USEPA) as 0.003 mg L^{-1} and 0.005 mg L^{-1}, respectively (Pyrzynska, 2019). Hence, the introduction of innovative green-friendly approaches for monitoring trace levels of cadmium with high accuracy is becoming increasingly crucial.

In the literature, several analytical techniques such as stripping voltammetry (Ma et al., 2021), graphite furnace atomic absorption spectrometry (GFAAS) (Saremi et al., 2025), UV-Vis spectrophotometry (Pawar et al., 2025), inductively coupled plasma-mass spectrometry (ICP-MS) (Kafouris et al., 2024), inductively coupled plasma-optical emission spectrometry (ICP-OES) (Khunou et al., 2024) and flame atomic absorption spectrometry (FAAS) (Soylak et al., 2024) have been developed for the determination of Cd in different matrices. FAAS is a simple, easy-to-operate, low-cost and fast technique with a simple interface that is widely used in many research laboratories. The low efficiency of the nebulizer unit is known to be the major disadvantage of the FAAS system. This is an important limitation for trace analyte determination in complex matrices (Kaya and Yaman, 2008). Additionally, interference effect due to the complexity of the sample matrix is the other challenge for the evaluation of measurements at low detection limits (İnce et al., 2024). On the other hand, the fact that real sample matrices often contain potential interferences requires the preconcentration and purification of target analytes prior to analytical measurements (Aziziyan et al., 2025). For this reason, a sample preparation step prior to analysis becomes important for efficient separation of the analyte(s) from the matrix. For this goal, several microextraction methods have been reported for the separation/preconcentration of Cd such as dispersive liquid-liquid microextraction (DLLME) (Samanpong et al., 2024), solidified floating organic drop microextraction (SFODME) (Alahabadi et al., 2017), hollow fiber liquid phase microextraction (HF-LPME) (Chen et al., 2014), cloud point microextraction (CPE) (Zolfaghari et al., 2024), and dispersive solid phase extraction (DSPE) (Rajabi et al., 2020) in recent years. Among them, DSPE is a popular approach that allows an efficient and rapid extraction process through direct contact of the sorbent material with the sample solution. Depending on the magnetic properties of the adsorbent, phase separation is achieved by an external magnetic field, centrifugation or filtration step. Then, the analytes adsorbed on the sorbent surface are desorbed by the addition of a suitable desorption solvent (Emmanuel Ibukun et al., 2024). DSPE also has the advantages of easy applicability, rapid extraction, high preconcentration factor, environmental friendliness and consistent with different analytical instruments and can be employed in the determination of various organic/inorganic analytes. In addition, the diversity in some properties of sorbents such as surface area, thermal, magnetic mechanical, catalytic and electronic properties The diversity of properties of sorbents including surface, magnetic, thermal, and electronics properties enhances the applicability/appeal of the DSPE method. (Khan et al., 2025). While metal oxide-based (Al Yaqoob et al., 2019), polymer-based (Panjali et al., 2015), and carbon-based (Zhang, 2025) nanostructures exist in literature for the preconcentration of Cd, these sorbents involve complex synthesis procedures. On the other hand, the application of waste materials in chemical processes after they have been reactivated introduces innovation to research. In recent years, waste toner particles have attracted interest in different fields as reusable beneficial materials (Behera et al., 2024; Du et al., 2025; Panchal et al., 2024). Toner particles are expected to be a good alternative for the preconcentration of organic/inorganic pollutants due to their attractive physical and chemical properties (Jamali et al., 2024). The reported studies in the literature have addressed the magnetic behavior attributed to components such as magnetite (Fe_3O_4) and other iron oxides, which enable toner separation under a magnetic field (Farajzadeh and Mohebbi, 2018; Yu et al., 2022). Due to these properties, toner particles may be potential materials for magnetic solid-phase extraction applications. In particular, the development of high-throughput methods is becoming increasingly important as monitoring trace contaminant levels in food for human consumption may require a preconcentration step.

In the literature, some efficient metal enrichment methods based on micro scale extraction have been reported using carbon-based or bio-based sorbents, characterized by low sorbent amounts, low sample volumes, and consistent with green chemistry principles (Karatepe and Soylak, 2014; Krawczyk and Jeszka-Skowron, 2016; López-García et al., 2019).

Cinnamon is a flavored herb that is growing in popularity due to its many health benefits, particularly its ability to lower blood glucose, serum lipids and insulin effectiveness associated with metabolic syndrome (Bibi et al., 2024). Cinnamon is known to reduce the risk of hyperglycemia, hyperlipidemia and cancer due to the presence of active functional compounds such as cinnamaldehyde, cinnamyl alcohol, cinnamyl acetate, eugenol, methyl hydroxy chalcone polymer, tannin, camphor and cinnamic acid (Senevirathne et al., 2022). For this reason, the detection of possible exposure to heavy metal contamination through roots, trunks, and leaves is very critical for human health. On the other hand, the environmental conditions of tea plant cultivation and the organic/inorganic compounds contained in tea plant are major factors affecting sensitive cadmium determinations. It is quite possible for inorganic pollutants such as cadmium from environmental sources to enter the plant, so the issue of monitoring trace levels allows for novel studies (Hocaoglu-Ozyigit and Nazli Genc, 2020). Furthermore, no previous study has focused specifically on the determination of cadmium at trace levels using selected solid phase extraction strategy in cinnamon or similar matrices.

This study involves the DSPE method in which toner particles were used for the first time as a sorbent for the preconcentration of Cd in any type of sample. Toner particles obtained from cartridge waste were used as sorbents for the determination of trace levels of cadmium in cinnamon tea samples, an aromatic spice frequently consumed by people in daily life, and the extraction conditions were univariately optimized to achieve high extraction efficiency. The fact that a preconcentration study for the determination of trace Cd based on toner particles has not been previously presented in the literature supports the novelty of the study. The recovery results obtained with the matrix matching calibration strategy and the waste toner-based DSPE-FAAS method were reported to be suitable and practical for the accurate and sensitive determination of critical levels of Cd reported by the WHO and USEPA.

2. Materials and methods

2.1. Reagents and chemicals

All experimental studies were performed using analytical grade chemicals and reagents. The waste toner particles were supplied from an expired cartridge compatible with the Canon printer. Cadmium sulphate-8/3 hydrate salt from Riedel-de Haen (98 %, Austria) was dissolved in ultrapure water to prepare main stock solution and the and working solutions and the calibration standard solutions were obtained at different concentrations with diluting the stock solution The concentrations of potassium hydrogen phthalate (Merck, Germany), hydroxymethyl aminomethane (tris) (Merck, Germany) and sodium tetraborate decahydrate (Sigma Aldrich, Germany) salts used for the preparation of buffer solutions between pH 4.0 and 9.0 were 0.40 M, 0.50 M and 0.06 M, respectively. Sodium hydroxide (NaOH) and 37 % of hydrochloric acid (HCl) with 0.10 M of concentrations used for pH adjustment and 65 % of nitric acid (HNO_3) used as elution solution were purchased from Merck, Germany. Acetylene gas bought from a local supplier in İstanbul, Türkiye was utilized as fuel source in FAAS system.

2.2. Instrumentation

A flame atomic absorption spectrophotometer (FAAS) model ATI UNICAM AA929 was employed for the determination of cadmium ions throughout the method development. The spectroscopic measurements were performed using a Cd hollow cathode lamp that produces light at a wavelength of 228.8 nm, a slit width of 0.50 nm and a current of 12 mA, and a deuterium (D_2) lamp integrated in the instrument for background corrections. Nuve FN120 oven (Türkiye) and Shimadzu (Japan) ATX224R precision balance were used for the drying and weighing of toner particles, respectively. The pH of the solutions was measured with a Mettler Toledo (USA) S220-K Seven Compact benchtop pH meter. Homogeneous mixing of the solutions was achieved utilizing an Isolab-M101002 model vortex mixer (Germany), MRC-Clean01 model ultrasonic bath and Heidolph (Germany) mechanical shaker. A neodymium purchased from an electronic market was used an external magnetic field to ensure the phase separation.

2.3. Preparation of toner particles

The toner particles used as sorbents throughout the study were easily collected from the cartridge reservoir of the printer in our laboratory. The washing step was achieved with ethanol and pure water to remove the possible impurities from the surface of toner particles. After the removal of the moisture in oven at 50 °C for 24 h. The dried particles were ground in a mortar to obtain a homogeneous particle size before using in the process.

2.4. Samples

Cinnamon tea, which is used to test the feasibility of the method developed in the study, is one of the spice teas that have a sweet and aromatic structure in daily life. Firstly, cinnamon samples of two different brands were obtained from the markets in the form of sticks. One cinnamon stick sample from each brand was reduced to a smaller size with a weighing amount of 1.0 g and placed in 200 mL of boiled water sample for brewing as for human consumption. Then, filtration was performed with the help of filter paper to separate the cinnamon particles from the liquid phase. After 20-fold dilution of the filtrate, the diluted solutions used as blank were spiked to a final Cd concentration of 5.0, 10, 15 and 20 μg L^{-1}.

2.5. Toner particles based DSPE-FAAS procedure

The DSPE-FAAS procedure based on toner particles developed for the determination of trace levels of Cd in cinnamon tea samples consists of buffering the sample solution and adding the sorbent material, adsorbing Cd ions by stirring the sorbent material, releasing Cd ions with a suitable desorption solvent after phase separation and performing measurements in the FAAS system. To summarize, the 24 mL of sample solution in a falcon tube was buffered by adding 0.50 mL of pH 8.0 solution and then 45 mg of toner particles were added as sorbent into the solution. For the adsorption of Cd ions by effective dispersion of toner particles without agglomeration in solution, ultrasonication for 7.5 min followed by vortexing for 15 s was applied. Then, the phase separation was carried out with the help of an external magnet to collect the toner particles at the bottom of the tube and ensure the easy decantation. Toner particles were mixed with 100 μL of 1.0 M of HNO_3 to release the Cd ions. The extraction phase was sent to FAAS system to determination of Cd.

3. Results and discussion

Optimization studies of the toner particle-based DSPE-FAAS method developed for the determination of cadmium ions at trace levels with high sensitivity and accuracy were carried out univariately to achieve the highest extraction efficiency. For this goal, the investigation of the optimum condition of any parameter was carried out by keeping the other variables in constant conditions. The optimum conditions were decided by considering the highest signal to noise ratio and repeatability in the responses obtained.

3.1. Characterization studies

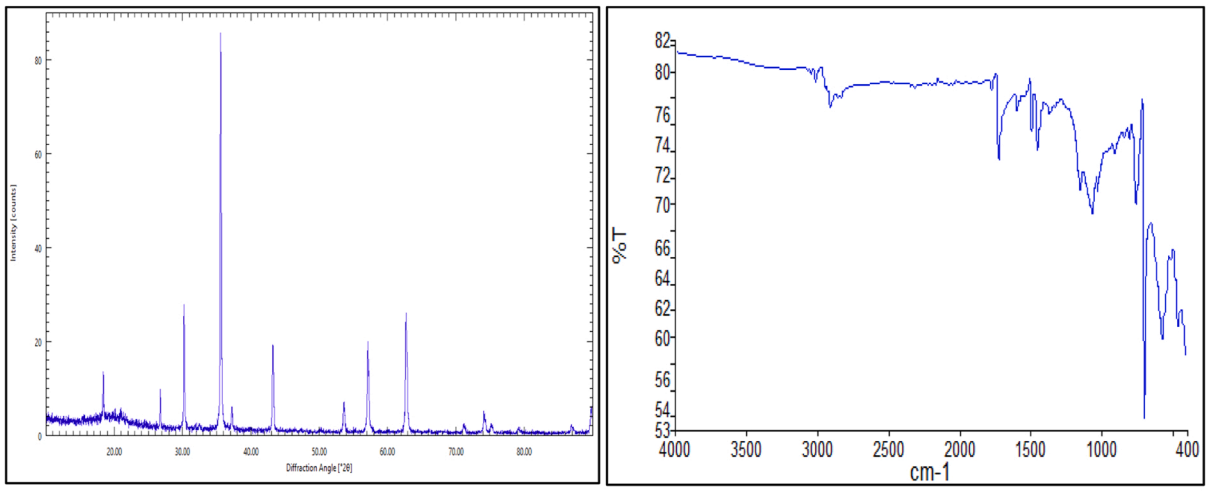
The characterization studies were performed by x-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) scanning electron microscopy (SEM), zeta potential and energy dispersive X-ray (EDX) techniques to investigate the crystalline structures, chemical properties of surface, morphological features, particle zeta potential distribution and elemental composition analysis, respectively.

In Fig. 1a, XRD results were presented with Cu-K α radiation with a wavelength of 1.54059 Å by scanning in the angular range 2.0–90° of 2θ with a step size of 0.013°. According to the 2θ degrees, the main phase of toner particles was iron oxide (Fe_3O_4). Briefly, 2θ degrees of 18.3, 30.1, 35.4, 37.1, 43.1, 53.4, 56.9 and 62.7 were in accordance with the Fe_3O_4 phase (JCPDS#19–0629) and correspond the lattice parameters (111), (220), (311), (222), (400), (422), (511) and (440), respectively (Nezhadi et al., 2020). The other peaks considered to be possible organic residues. In Fig. 1b, FT-IR spectrum was shown by scanning the particles for the assessment from 400 cm^{-1} to 4000 cm^{-1}. The peak recorded near 2947 cm^{-1} is evidence of the presence of the C-H stretch, which is commonly observed in the 2918–2954 cm^{-1} range (Francis Xavier et al., 2022) Moreover, the peak at 696 cm^{-1}, 1450 cm^{-1}, 1723 cm^{-1} and 3023 cm^{-1} correspond to C-Cl stretch in alky halides, C-H bend in alkanes, C═O stretch in saturated aliphatic, esters and C-H aromatic respectively (Udoji Itodo et al., 2018). Figs. 1c and 1d show the surface morphology of toner particles with different magnitudes. In Fig. 1e, the zeta potential was determined −8.2 mV under the conditions 1041.7 of count rate (kcps) and 24.9 °C. In Figure1f, elemental analysis of toner particles was presented and carbon (C), iron (Fe), oxygen (O) and silicon (Si) were detected by EDX technique.

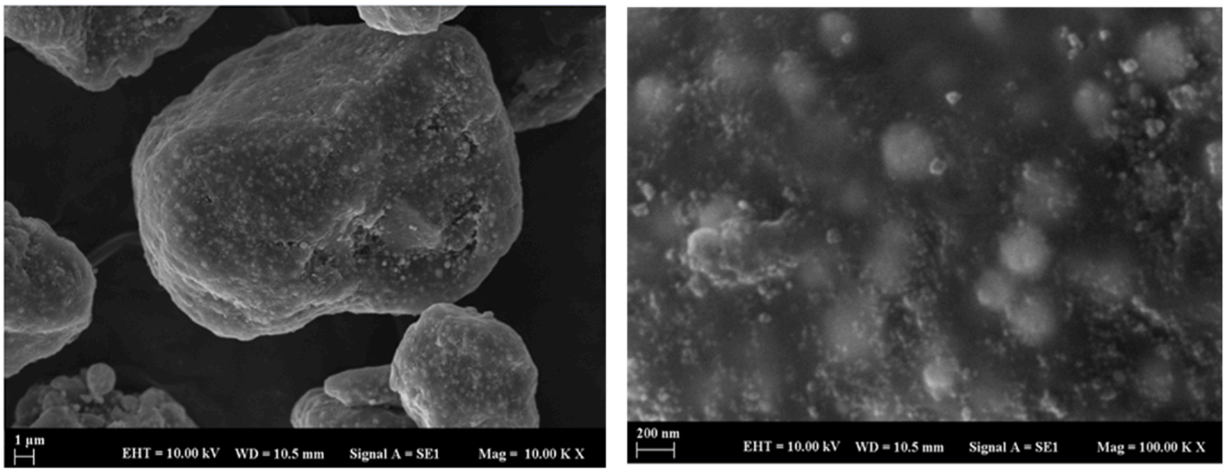
3.2. Influence of pH and volume of Buffer

The pH of the sample solution is an important parameter affecting DSPE outputs for the adsorption of metal ions at the interface between aqueous media and adsorbent. Because the pH is highly effective on the chemical form of the binding sites of the sorbent, the competitive adsorption between heavy metal ions and hydronium ions, and the type of bonds formed (Sürme et al., 2024). In parallel, while Cd^{2+} ions remain in the free form in solution under acidic conditions, the surface groups on the sorbent are in the protonated form. Under alkaline conditions, Cd^{2+} ions can precipitate as hydroxide forms in the solution, while the surface functional groups are deprotonated. An increase in the pH value of aqueous samples results in the surface charge of magnetic particles becoming increasingly negative and positively charged Cd^{2+} ions being adsorbed more strongly onto the particle surface (El-Sheikh et al., 2023). The pH values of the solution between pH 4.0 and 9.0 were tested to evaluate their effect on the preconcentration of Cd^{2+} ions. In the results obtained (Figure S1), the mean absorbance values increased slightly from pH 4.0–7.0, increased sharply from 7.0 to 8.0 and were recorded as close values between pH 8.0 and 9.0. Since it is indicated in the literature that Cd in the aqueous media starts to precipitate in hydroxyl form after pH 8.0, pH 8.0 was selected as the optimum pH of the sample solution Moreover, in alkaline region, increasing the pH value allows the amount of negatively charged sites on the sorbent surface and the extraction efficiency of Cd^{2+} may have increased due to electrostatic interaction with the magnetic properties of toner particles (İnce et al., 2024).

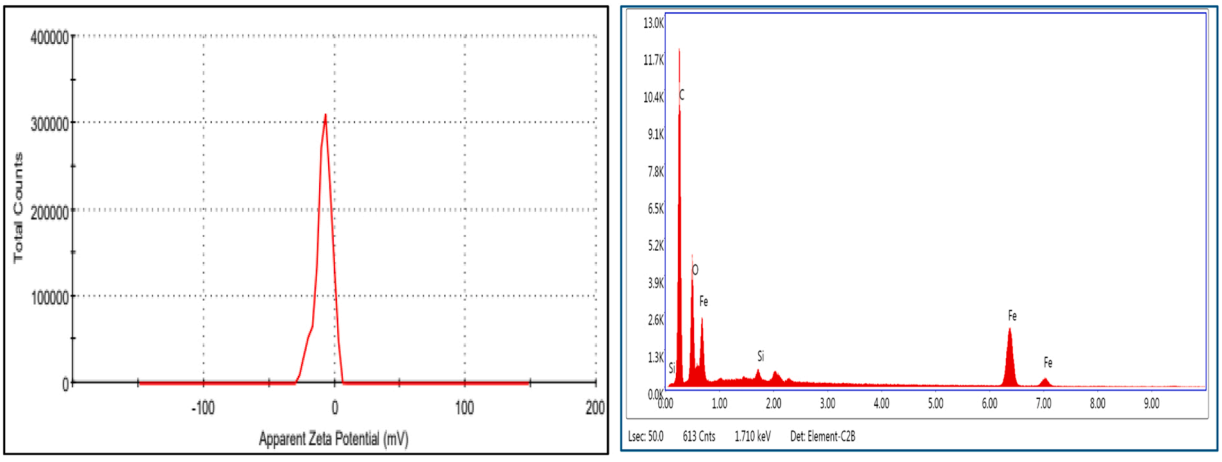
Different buffer solution volumes between 0.50 and 2.5 mL were added to the sample solution in 0.50 mL increments and the extraction procedure was performed under equal conditions. The highest mean



(a) (b)



(c) (d)



(e) (f)

Fig. 1. The characterization results of toner particles a) XRD image b) FT-IR image c) SEM image with 1.0 µm scale d) SEM image with 0.20 µm scale e) Zeta potential f) EDX image.

absorbance values were recorded at 0.50 mL and decreased with sample dilution from 0.50 mL to 2.5 mL (Figure S2). For this reason, 0.50 mL was selected as the optimum buffer volume for the pH 8.0.

3.3. Influence of sorbent amount

The amount of sorbent material is an important variable in the evaluation of extraction efficiency. The reason is that inadequate amounts of sorbent used for the extraction of a certain amount of analyte ions will lead to poor extraction outputs (Afshar Mogaddam et al., 2025). Moreover, an excessive amount of sorbent would lead to unnecessary chemical usage, which would increase both sorbent consumption and operating costs, contradicting the principles of green chemistry (Altunay et al., 2022). Various sorbent amounts between 15 and 55 mg in 10 mg increments were tested to investigate the adsorption ability on the preconcentration of Cd^{2+} ions. According to the results shown in Fig. 2, the mean absorbance values were close between 15 and 35 mg and increased slightly from 25 mg to 55 mg. The highest extraction outputs were recorded for 55 mg, but 45 mg was chosen as the optimum toner particle amount due to high repeatability and it was predicted that solvent consumption for elution and sorbent requirement would increase. Moreover, a larger amount of toner particles may allow to adsorb more analyte, but requiring a higher volume of eluent for the desorption will take the method out of the concept of green chemistry.

3.4. Influence of sample volume

Sample volume, which directly affects extraction efficiency, is one of the main factors to be evaluated, especially in terms of the transfer of low concentrations of analytes to the sorbent surface. Although it is considered useful to work with low volumes of sample matrix to reduce extraction period and possible matrix effects, it is necessary to increase the sample volume for a constant final volume to achieve high extraction efficiency (Maranata et al., 2021). For this objective, the extraction process was carried out under equal experimental conditions using initial volumes of 8, 16, 24, 32 and 40 mL to determine the optimum sample volume. In the measurement results shown in Fig. 3, it was recorded that the mean absorbance values increased linearly from 8.0 mL to 24 mL and were very close to each other between 24 mL and 40 mL. The fact that extraction outputs between 24 mL and 40 mL were close to each other is related to the limited sorbent surface despite the increase in the amount of analyte ions in the sample medium or insufficient amount of desorption solvent. Hence, 24 mL was determined as optimum initial sample volume.

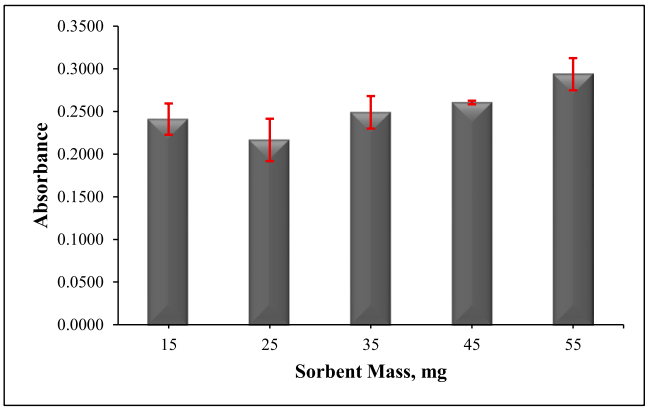


Fig. 2. Influence of sorbent mass on extraction outputs (40 mL of 0.025 mg L^{-1} of Cd^{2+} solution, 0.50 mL of pH 8.0, 300 s ultrasonication + 30 s vortex, 200 μL of conc. HNO_3) (n = 3).

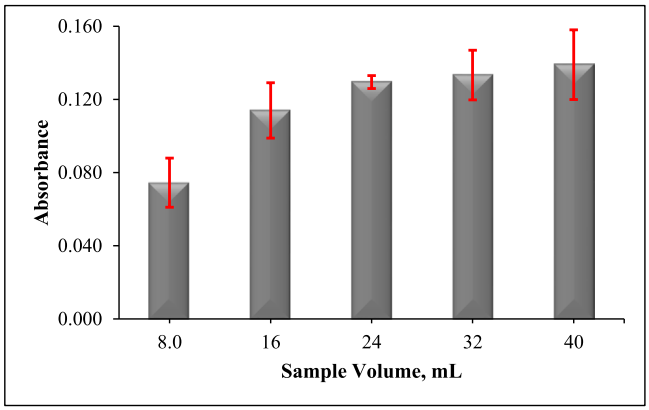


Fig. 3. Influence of sample volume on extraction outputs (0.50 mL of pH 8.0, 45 mg of sorbent, 300 s ultrasonication + 30 s vortex, 200 μL of conc. HNO_3) (n = 3).

3.5. Influence of mixing type and period

Mixing process is necessary to increase the interaction of the analyte included in the sample solution with the sorbent and for the efficient transfer to the sorbent surface. The agglomeration of toner particles is one of the most important concerns for extraction efficiency in the developed method. Because the tendency of the particles to agglomerate will reduce the total surface area, low extraction outputs are likely to occur. In the preliminary experiments, the effect of agglomeration was compared between direct vortex mixing after adding toner particles to the solution and vortex mixing after ultrasonication. The results obtained showed that the ultrasonication step is important for the improvement of the dispersion properties of the toner particles. For this reason, the toner particles added to the sample solution were subjected to ultrasonication for 1.0, 2.5, 5.0, 7.5 and 10 min before mixing and the extraction process was carried out under equal conditions with vortex and the mean absorbance values recorded increased from 1.0 min to 7.5 min and were very close for 7.5 and 10 min (Figure S3). Therefore, 7.5 min was determined as the optimum ultrasonication period. Then, different mixing types such as vortex, orbital shaker, manual shaking and ultrasonication were applied to evaluate the optimum mixing type. Based on the mean absorbance values, it was observed that the vortex enriched the interaction between analyte and adsorbent 25 % more than the other 3 mixing types (Figure S4). For vortex, which was determined as the optimum mixing type, extraction outputs at application periods of 15, 30, 45 and 60 s were evaluated. The all recorded mean absorbance values were close each other (Figure S5). Hence, 15 s was chosen as optimum vortexing period.

3.6. Influence of desorption solvent concentration and volume

In the DSPE method, the analyte(s) transferred to the sorbent surface and separated from the solution medium are desorbed from the sorbent surface with the help of a suitable solvent. For this purpose, acid solutions that generate the protonation at the active sites of the sorbent surface are often used in the desorption step. Nitric acid, which is one of the protic solvents, is widely utilized with the features the no interference effects in analysis and achievement the high extraction outputs (Timur et al., 2009). First, different concentrations of HNO_3 between 0.50 and 14.4 M were utilized to elute the Cd^{2+} ions from the surface of toner particles. The recorded mean absorbance values showed an increase from 0.50 M to 1.0 M and close to each other between 1.0 and 14.4 M. Hence, 1.0 M was selected as optimum HNO_3 concentration. Then, the eluent volume, which is the critical parameter to determine the high preconcentration factor as final volume, was optimized by employing 100, 125, 150 and 200 μL of 1.0 M of HNO_3. As shown in

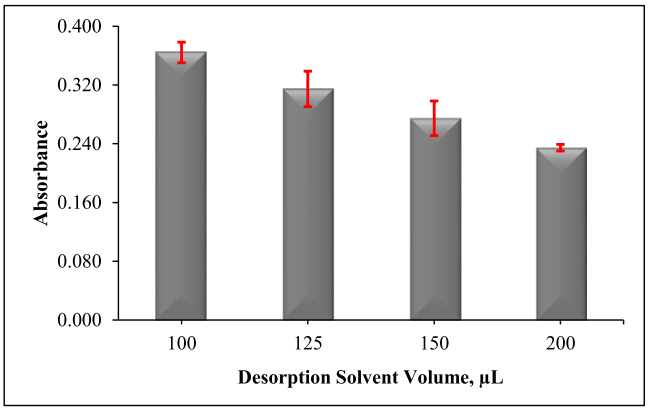


Fig. 4. Influence of eluent volume on extraction outputs (24 mL of 25 µg L^{-1} Cd^{2+} solution, 0.50 mL of pH 8.0, 45 mg of sorbent, 7.5 min ultrasonication + 15 s vortex) (n = 3).

Fig. 4, the highest mean absorbance value was recorded for 100 µL and a linear decrease was observed from 100 µL to 200 µL due to dilution of Cd^{2+} ions. In preliminary tests, lower volumes than 100 µL was not sufficient for the collection and feed the extraction phase to the instrument. For this reason, 100 µL was determined as optimum HNO_3 volume.

3.7. Analytical figures of merit

The analytical performance developed toner-based DSPE-FAAS system for Cd^{2+} determination were assessed in terms of limit of detection (LOD) / quantification (LOQ), linear working range, regression coefficient (R^2) and percent relative standard deviation (%RSD) under optimum conditions determined as 24 mL of sample, 0.50 mL of pH 8.0, 45 mg of sorbent, 7.5 min ultrasonication + 15 s vortex mixing and 100 μL^{-1} of 1.0 M of HNO_3. In the calculation of LOD and LOQ, the standard deviation of at least five replicates of the lowest concentration of the calibration plot was multiplied by 3 and 10, respectively, and divided by the slope of the linear equation of the calibration plot and calculated as 0.55 and 1.82 µg L^{-1}, respectively. A good linearity in the range between 1.5 and 40 µg L^{-1} was achieved with the R^2 of 0.9995. Furthermore, the %RSD for 1.5 µg L^{-1} was found to be 7.2 %. The calibration plot equations in units of mg L^{-1} were $y = 0.2127x + 0.0015$ and $y = 8.6952x + 0.0109$ for FAAS and toner-based DSPE-FAAS, respectively. The increase in sensitivity was calculated as 40.9 by comparing the ratio of the slopes of the calibration plot obtained for the DSPE-FAAS and FAAS systems, respectively.The analytical figures for both systems are presented in Table 1.

The analytical performance values of developed waste toner particles based DSPE-FAAS method for determination of Cd^{2+} ions are compared with other DSPE methods in the literature in Table 2. Briefly, Melo et al. used 100 mg of 2-(2-thiazolylazo)-5-dimethylaminephenol loaded Amberlite XAD-2 resin as sorbent material for the separation of Cd^{2+} ions and reported 108-fold improvement in the detection power with a LOD of 1.2 µg L^{-1} by FAAS with continuous flow assisted SPE (Melo et al., 2000). El Sheikh et al. utilized 50 mg of citric acid coated magnetic carbon nanotube nanoparticles as a sorbent to preconcentrate the Cd^{2+} ions in well and tap water samples by 10 times and declared the LOD value as 1.8 µg L^{-1} (El-Sheikh et al., 2019). Yılmaz et al. reported a vortex-assisted DSPE method to trace determination the Cd^{2+} ions in soft drink, water and some food samples utilizing 250 mg of magnetic polystyrene-b-poly dimethyl siloxane hydrophobic block copolymer and achieved a LOD of 1.7 µg L^{-1} with a 50-fold increase in sensitivity (Yılmaz et al., 2023). Soylak et al. reported a DSPE method for the preconcentration of Cd^{2+} ions in tea, water, chocolate, spice, and tobacco samples using a magnetic metal-organic framework based Fe_3O_4-SiO_2-MIL-53 (Fe) nanocomposite. LOD was recorded as 1.3 µg L^{-1} (Soylak et al., 2023). As seen, each of these methods involves long and complex procedures to fabricate the sorbents. Moreover, considering the operating cost and the labor of the analyst, the toner particles used in the developed method were easily obtained from the cartridge reservoirs separated as waste without any labor or cost, and offer a novel approach that can compete with other methods with a satisfactory preconcentration factor of 40.9 times.

3.8. Assessment of developed method in real samples

Recovery tests are highly important for verifying the feasibility of a developed/proposed analytical protocol. In this context, the applicability of the waste toner-based DSPE-FAAS method developed in this study was investigated by spike tests for different concentrations of cinnamon tea samples, which are consumed by humans and whose benefits are described in the "Introduction" section. Before spike experiments, sample analysis was performed to investigate the presence of Cd^{2+}. However, since no analytical signal was observed, the concentrations of 5.0, 10, 15 and 20 µg L^{-1} were spiked considering the linear calibration range. Then spectroscopic measurements were carried out to assess the recovery results. The measured absorbance values were converted to concentration using the calibration plot equation obtained with aqueous Cd^{2+} standards. The findings given in Table 3 showed that the recovery results between 71.5 % and 98.7 % calculated by external calibration were acceptable. On the other hand, a matrix matching calibration strategy was also used to minimize the possible interference effects caused by the matrix and improve the accuracy of measurement results. For this purpose, two different calibration plots were drawn for each tea sample, and the % recovery results for each tea sample was calculated utilizing the calibration plot equation of the other sample. The recovery results calculated between 90.4 % and 119.0 % with the matrix matching calibration strategy demonstrated that the developed waste toner based DSPE-FAAS method is feasible for the determination of trace levels of Cd^{2+} ions in cinnamon tea samples with high accuracy and sensitivity.

4. Conclusion

In this research, an innovative preconcentration method based on DSPE for the determination of trace levels of cadmium, which has toxic effects on human health and ecosystems, has been presented. For this purpose, toner particles with magnetic properties obtained from printer cartridges discarded from printing and copying activities were used as sorbent material in the analytical protocol developed with the principle of converting waste materials into usable and beneficial products. The re-functionalization of waste material allows the elimination of criteria such as cost, time, chemical consumption and labor required for the synthesis of sorbent material, thus developing a method in accordance with the principles of green chemistry. The parameters that play an important role on the extraction efficiency were optimized by univariate optimization approach and the LOD was calculated to be 0.55 µg L^{-1}.

Table 1
The system analytical performance values of the FAAS and toner based DSPE-FAAS.

Method	LOD, µg L^{-1}	LOQ, µg L^{-1}	R^2	Linear Range, µg L^{-1}	Linear Equation (mg L^{-1})	Enhancement in sensitivity
FAAS	41.1	136.9	0.9988	100–4000	$y = 0.2127x + 0.0015$	-
Toner based DSPE-FAAS	0.55	1.8	0.9995	1.5–40	$y = 8.6952x + 0.0109$	40.9

Table 2
Evaluation of literature findings on SPE-FAAS methods.

Sorbent	LOD, μg L^{-1}	Linear Range, μg L^{-1}	Interaction Period	Sorbent Amount, mg	Eluent volume, mL	EF/PF *	Related work
Amberlite XAD-2 resin loaded with 2-(2-thiazolylazo)-5-dimethylaminephenol (TAM)	1.2	0 – 200	1.0 min	100	2.5	108	(Melo et al., 2000)
Citric acid coated magnetic carbon nanotube	1.8	6.1 – 300	30 min	50	10	10	(El-Sheikh et al., 2019)
Magnetic polystyrene-b-poly dimethyl siloxane hydrophobic block copolymer	1.7	5.1 – 100	10 min	250	1.0	50	(Yılmaz et al., 2023)
Fe_3O_4-SiO_2-MIL-53 (Fe) nanocomposites	1.3	4.3–500	4.0 min	20	4.0	4.9	(Soylak et al., 2023)
Waste toner particles	0.55	1.5 – 40	7.75 min**	45	0.10	40.9	This work

* EF/PF: Enhancement/preconcentration factor
** 7.5 min ultrasonication + 15 s vortex

Table 3
Recovery calculations of cinnamon tea matrices utilizing external and matrix matching calibration (n = 4).

	Spike Concentration, μg L^{-1}	External Calibration Recovery ± SD, %	Matrix Matching Calibration Recovery ± SD, %
SAMPLE 1	0 (Sample)	ND	ND
	5.0	89.3 ± 11.8	119.0 ± 11.4
	10	85.2 ± 4.1	98.7 ± 4.0
	15	89.5 ± 7.5	97.4 ± 7.3
	20	98.7 ± 14.1	103.6 ± 13.6
SAMPLE 2	0 (Sample)	ND	ND
	5.0	71.5 ± 9.9	91.3 ± 9.6
	10	82.0 ± 17.5	90.4 ± 16.9
	15	96.4 ± 9.2	100.6 ± 8.9
	20	93.6 ± 9.8	96.0 ± 9.5

*SD: Standard Deviation, ND: Not Detected

The sensitivity of the conventional FAAS system was improved by a factor of 40.9. The applicability of the proposed method was investigated by spike tests on different cinnamon tea samples. The recovery results between 90.4 % and 119 % obtained by matrix matching showed that the waste toner-based DSPE-FAAS method is feasible and applicable for sensitive and accurate Cd determination at critical levels reported by WHO and USEPA. The developed method is expected to shed light on innovative studies in the field of green chemistry, not only for converting waste into useful products, but also for the removal or trace-level determination of various organic/inorganic pollutants.

CRediT authorship contribution statement

Sezgin Bakırdere: Writing – review & editing, Supervision, Methodology, Data curation, Conceptualization. **Hakan Serbest:** Writing – original draft, Visualization, Validation, Methodology, Formal analysis, Data curation. **Ahsen Bayraktar:** Writing – original draft, Visualization, Validation, Formal analysis. **Muhammed Ali Büyük:** Writing – original draft, Visualization, Validation, Formal analysis. **Büşra Ali:** Writing – original draft, Visualization, Validation, Formal analysis.

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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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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jfca.2025.108636.

Data availability

Data will be made available on request.

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