

Application of response surface methodology for optimization and determination of nickel by syringe-to-syringe dispersive liquid phase micro-extraction in environmental water and herb samples coupled with flame atomic absorption spectrometry

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Abstract

A novel, rapid, simple and sensitive syringe-to-syringe-dispersive liquid-phase micro-extraction (SS-DLPME) method followed by flame atomic absorption spectrometry was proposed for pre-concentration and determination of nickel ion in water and herbal samples using 1-(2-pyridylazo)-2-naphthol as a chelating agent. Various experimental factors on pre-concentration and determination of nickel such as pH of sample solution, concentration of the complex agent, volume of the extraction solvent, shooting times, centrifugation time and ionic strength were investigated using Plackett–Burman design for screening and Box-Behnken design as an optimization method. Under optimum conditions, the calibration curve was linear over the range of 10– 875 $\mu\text{g L}^{-1}$ with the coefficient of determination $R^2 = 0.997$ and the detection limit of 1.800 $\mu\text{g L}^{-1}$. Relative standard deviation (R.S.D) for 8 replicate determinations and the enrichment factor (EF) were 2.050% and 86, respectively. Finally, the proposed method was applied successfully to pre-concentration and determination of the analyte in environmental water and herbal samples.

Keywords: Multivariate optimization; nickel; syringe to syringe dispersive liquid – phase micro-extraction; water and herb samples.

Introduction

Nickel is a heavy metal and a moderate toxic element in the Earth's crust. The pollution of the environment by toxic elements and heavy metals has received important attention recently. Most heavy metals persist in the environment and cause pollution in air, soil, and water ecosystems [1] and these cations considered as a hazardous class of environmental pollutants can be harmful to human health even at low concentrations [2]. Nickel and its

compounds can lead to serious problems, including allergic reactions, respiratory system cancer [3] and certain nickel compounds may be carcinogenic [4]. Thus, sensitive methods are needed to detect this metal in most environmental samples. Several analytical techniques, such as GFAAS [5], flame-AAS [6], ICP-AES [7] and ICP-MS [8] have been applied to accurately determine trace amount of nickel in various samples. AAS is comparatively an inexpensive, robust

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and easy-to-operate analytical technique. The detection of trace nickel in aqueous samples is difficult because of particularly low concentration and matrix effects [9]. Therefore, pre-concentration techniques [10] adopted by various researchers based on chemical, physical and physicochemical principles. Several methods have been successfully applied to pre-concentration of trace Ni including: co-precipitation [11], liquid-liquid extraction (LLE) [12], ion-exchange [13], and solid-phase extraction (SPE) [14].

Recently, a novel mode of DLPME (dispersive liquid-phase micro-extraction) called SS-DLPME (syringe-to-syringe dispersive liquid phase micro-extraction). Asadi et al., in 2016, developed DLPME using a syringe-to-syringe system [15]. In syringe-to-syringe dispersive liquid-phase micro-extraction method (SS-DLPME), two syringes are connected to each other through one needle. The first syringe holding the aqueous sample and the extraction solvent is connected to the second syringe. The extraction solvent is dispersed in the aqueous sample through the consecutive injections between these two syringes until the extractant is thoroughly mixed with aqueous sample. Eventually, the mixture is transferred to a vessel, and the phases are separated by means of centrifugation.

In the present work, the SS-DLPME technique was developed for pre-concentration and determination of nickel in water and herbal samples. Chemometric tools have been applied to analytical method optimization [16].

Experimental

Chemicals and reagents

All the reagents used in this study were of analytical grade. The tested

extraction solvents, including cyclohexanol, 1-octanol, 1-decanol, 1-undecanol and 1-dodecanol were purchased from Merck (Darmstadt, Germany). Acetic acid (98%), hydrochloric acid (37%), sulphuric acid (98%), nitric acid (65%), ammonia, methanol, sodium hydroxide, sodium nitrate, nickel nitrate and sodium chloride (NaCl) were obtained from Merck (Darmstadt, Germany). The standard stock solution (1000 mg L^{-1}) of Ni (II) was prepared by dissolving appropriate amounts of nitrate salts in deionized water. A solution of 0.25 g L^{-1} 1-(2-pyridylazo) 2-naphthol (PAN) as the chelating agent (Merck, Darmstadt, Germany) was prepared by dissolving an appropriate amount of it in 1-octanol and lower concentrations were prepared by diluting the stock solution with 1-octanol.

Instruments and software

A flame atomic absorption spectrophotometer (Shimadzu AA-680, Japan) was equipped with a deuterium lamp and a background correction system was used for determination of Ni. Acetylene and air flow rate were 2.2 and 8.0 L min^{-1} respectively. A hollow-cathode lamp (Hamamatsu, Photonic Ni. Ltd, L233-series) at a wavelength of 232.4 nm was used as a radiation source. A Behdad Universal Centrifuge (Esfahan, Iran) was used as the phase separation in the SS-DLPME process. The pH adjustment of sample solution was done using a Metrohm pH-meter model 780 equipped with a glass-calomel electrode. The experimental design analysis was carried out with Minitab Version 16.

Sample preparation

The several water samples including tap, well, river and mineral waters were

tested. The water samples were collected from various regions of Malayer and Arak city. First, to remove any suspended material, the water samples were filtered with a 0.45 μm pore size membrane filter. Afterwards, the pH of water samples were adjusted at 9.7 by buffer solution and analyzed immediately.

Lettuce, spinach and tobacco samples were purchased from local supermarkets in Arak, Iran. At First, the collected samples were washed with distilled water for several times, and then dried at 100 $^{\circ}\text{C}$ for 24 h. A weight of 0.5 mg of them was weighted in porcelain crucible and transferred into the electrical furnace at 600 $^{\circ}\text{C}$ and maintained for 7 h. Afterward, the appropriate amount of hydrochloride solution (20% v/v) was added to digest ash powder. Then, the mixture was diluted by distilled water to 25 mL in volumetric flask [17]. The obtained solutions were used to analyze nickel contents of each sample by the proposed method.

SS-DLPME procedure

The extraction procedure steps are illustrated schematically in Figure 1. At first, the pH of 20 mL sample solution

containing 100 $\mu\text{g L}^{-1}$ of Ni (II) was adjusted at 9.7 and drawn into the syringe 1. Then, 150 μL 1-octanol (as the extraction solvent) containing 0.33 mg PAN was added into the sample solution by a 250 μL Hamilton gas-tight syringe (Figure 1-a). The needle of syringe 1 was removed and connected to syringe 2 using a silicone connector. After that, the solution in syringe 1 was rapidly injected into syringe 2, followed by back injection of the mixture into syringe 1 (Figure 1-b) for 15 times, until the extraction solvent was completely dispersed in the aqueous phase. Afterwards, the cloudy mixture was transferred into a 25.0 mL extraction vessel, and an appropriate volume of distilled water was added into the vessel until the solution reached to the top of the narrow neck section (Figure 1-c). Separation of the aqueous and organic phase was carried out by centrifuging for 10 min at 3500 rpm (Figure 1-d). Finally, the extraction phase was collected in Eppendorf tube by a 250 μL Hamilton gas-tight syringe, diluted to 300 μL with methanol and injected by a micro-injection system to the FAAS for Ni determination (Figure 1-e).

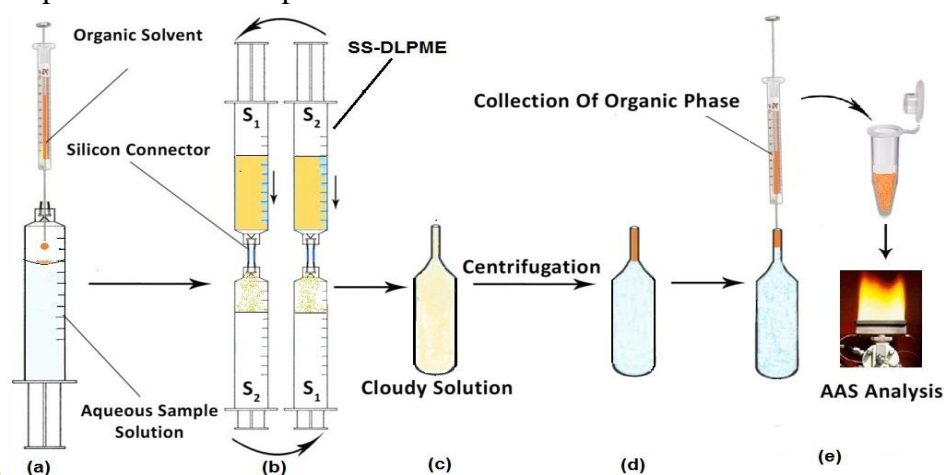


Figure1. Schematic procedure of the SS-DLPME method

Multivariate optimization

In this procedure, there are several factors which can affect the extraction

performance. Thus, a multivariate approach of experimental design is used for screening and optimization purpose. The Plackett–Burman design was used for screening the effective variables and then, in order to evaluate the significance of effective variables and investigate the interaction among them, a Box-Behnken design (BBD) was employed. To evaluate the significance of six variables including, volume of

the extraction solvent (X_1 : 75 – 300 μL), concentration of PAN (X_2 : 0.000016 – 0.00010 mol L^{-1}), pH of the sample solution (X_3 : 7.0 – 12.0), amount of salt (X_4 : 0 - 4%), shooting times (X_5 : 3 – 15) and centrifugation time (X_6 : 1 – 10 min), 22 experiments were performed with two center points in triplicate, and the mean of responses was evaluated. The variable and their levels are summarized in Table 1.

Table 1. Experimental variables and levels of the Plackett–Burman design

Variable	Key	Level	
		Low	High
Extraction solvent volume (μL)	X_1	75	300
Concentration of PAN (mg)	X_2	0.000016	0.0001
pH of the sample solution	X_3	7	12
Salt amount (% , w/v)	X_4	0	4
Shooting times	X_5	3	15
Centrifugation time (min)	X_6	1	10

After PBD, three significant variables (pH of the sample solution (X_1), concentration of PAN (X_2) and volume of the extraction solvent (X_3)) were selected and the Box–Behnken design (BBD) was applied to optimize and investigate the interactions between these significant parameters. The total number of experimental runs (N) in BBD is calculated by the following equation:

$$N=2K(K-1)+C_0$$

Where k is the variable number and C_0 is the number of center points. Therefore, 15 experiments were performed for optimizing these variables with three center point.

Results and discussion

Selection of the extraction solvent

The property of extraction solvent is an important parameter in the efficiency of micro-extraction techniques. The extraction solvent should have several

characteristics such as high extraction capability for the given analyte, lower density than water, low toxicity, low cost, no interference with analyte signal and compatibility with analytical instrument. In SS-DLPME method, the extraction solvent with low density was dispersed into the sample solution. Thereby, efficient interaction between sample medium and extraction solvent causes the mass transfer between two immiscible phases to enhance and leads to extraction efficiency in short equilibrium time. Therefore, to study the effect of the extraction solvent, several organic solvents including cyclohexanol, 1-octanol, 1-decanol, 1-undecanol and 1-dodecanol on the extraction of Ni by SS-DLPME method was studied. According to the obtained results, 1-octanol presented the highest absorbance and therefore, it was chosen for subsequent experiments (Figure 2).

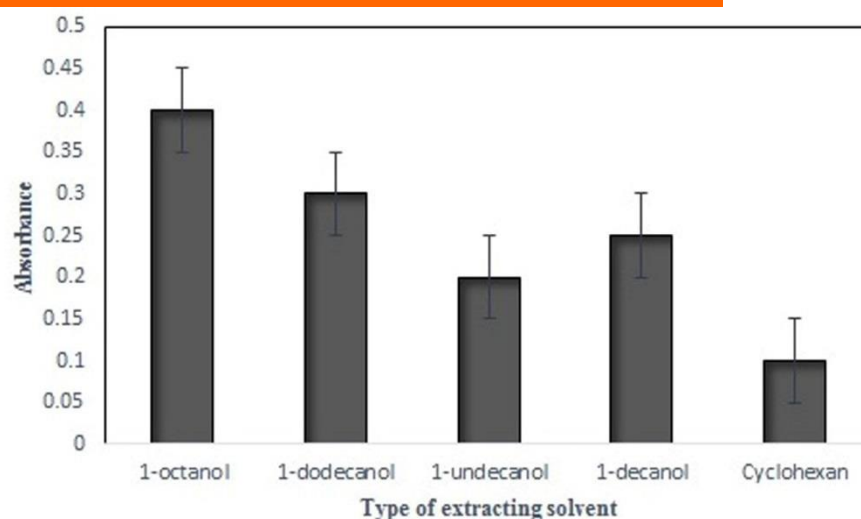


Figure 2. Effect of extraction solvent type on the extraction efficiency Ni^{2+} obtained from SS-DLPME. Extraction conditions: sample volume, 20 mL; amount of PAN, 0.33 mg ; pH, 9; extraction solvent volume, 150 μL ; number of injections, 5; and centrifugation time, 5 min. The error bars represent standard deviations ($n = 3$).

Optimization stage

Plackett–Burman design (PBD)

The Plackett–Burman design (PBD) is a useful two-level fractional factorial screening design. It is a very efficient screening design when only the main effects are of interest and is generated in steps from 4 to 100 runs [18]. In the present study, based on the preliminary experiments, six factors might have affected the experimental response. These factors are extraction solvent volume, concentration of PAN, pH of the sample solution, salt amount, and shooting times and centrifugation time. For this purpose, 22 experiments were performed randomly at two levels

and summarized in Table S1. The analysis of variance (ANOVA) results were evaluated for determination of the main effects. The effect of the factors was shown in Parato chart in 95% confidence level. According to Parato chart (Figure 3), extraction solvent volume, concentration of PAN and pH had the most significant effects and the salt effect, shooting times and centrifugation time had no significant effects in extraction efficiency. Thus, to continue the optimization with BBD, insignificant variables were kept constant in the center point values.

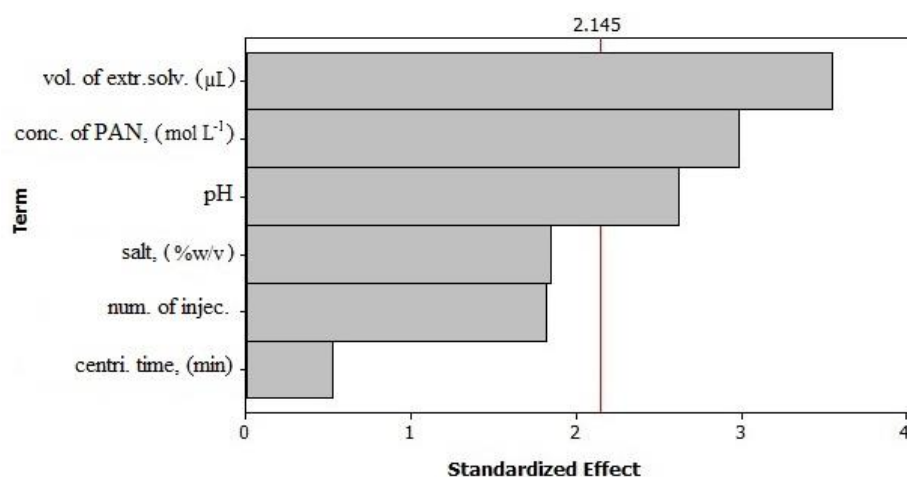


Figure 3. Pareto charts of the main effects obtained from the Plackett–Burman design ($\text{Alpha}=0.05$)

Box– Behnken design (BBD)

Box– Behnken design was employed to optimize the three factors (extraction solvent volume, concentration of PAN and pH of solution) that were chosen from the first screening design (PBD). In this step, 15 tests were performed and ANOVA was used in order to evaluate the statistical significance of the purposed method. The design matrix with the values of response is shown in Table S2. This design permitted the response to be modeled by a second-order polynomial fit, which can be expressed as the following equation:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_{12} + \beta_{22} X_{22} + \beta_{33} X_{32} \quad (1)$$

Where X_1 , X_2 , and X_3 are the independent variables, β_0 is the intercept, β_1 – β_{33} is the regression coefficients, and Y is the response function (Absorbance signal). The relationship between response and the three variables obtained by the application of response surface methodology (RSM) can be described with the following second-order equation:

$$Y = -4.16447 + 0.00414253 X_1 + 2.58199 X_2 + 0.832955 X_3 - 1.34683 \times 10^{-5} X_{11} - 3.27114 X_{22} - 0.0422333 X_{33} - 1.86047 \times 10^{-4} X_1 X_2 + 4.44444 \times 10^{-6} X_1 X_3 - 0.0372093 X_3 X_2 \quad (2)$$

Where Y is the response variable and X_1 , X_2 and X_3 are the coded values of volume of the extraction solvent, concentration of PAN, pH, respectively. The optimum conditions for extraction of Ni ions was obtained by solving the derivation from Equation 2 for the conditions of $\delta(A)/\delta(X_1) = 0$, $\delta(A)/\delta(X_2) = 0$ and $\delta(A)/\delta(X_3) = 0$, which were as follows: extraction solvent volume (X_1) = 152 μL , concentration of PAN (X_2) = 0.000066 mol L^{-1} and pH (X_3) = 9.73.

In ANOVA table, the P-value and lack of fit (LOF) are important parameters. If p-value is less than 0.05, then the model is statistically significant. The LOF parameter indicates the variation of signals around the fitted model. According to the obtained results, the p-value for lack-of-fit is 0.247 which confirms a good fitting of model to responses. The R^2 value shows that the calculated model could describe 0.9775 of the experimental results, the adjusted determination coefficient (R^2_{adj}) value is equal to 0.9371 and it also indicates an agreement between responses and the fitted model (Table S3).

In the design experiment software, there are some diagrams that can be applied to investigate the validity of the model. As can be seen in Figure 4a, random scatters evenly distributed above and below the horizontal axis prove the sufficiency of the model; also, according to Figure 4b, the normal probability diagram proves the distribution of errors with an average value of zero.

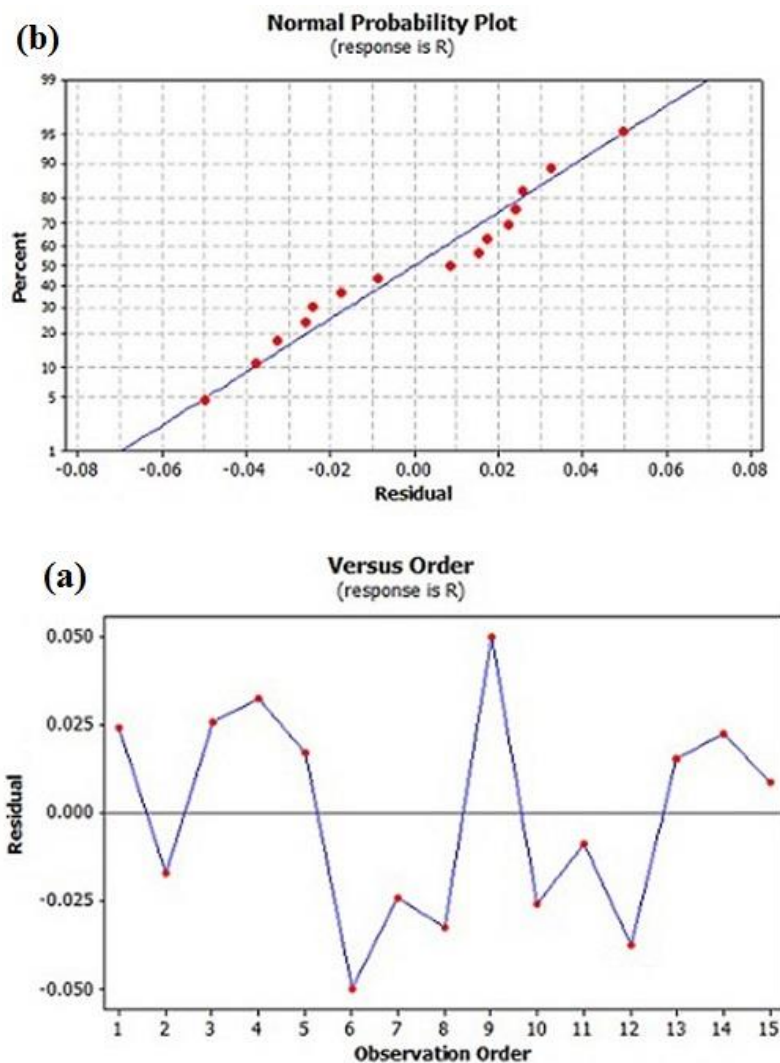


Figure 4. (a) Residuals vs. Run, (b) Normal probability of residuals

The conditions of the variables and evaluating the effect of various variables on the response are visualized by the three-dimensional response surface plots (Figures 5a-c). According to the response surface plot of Figure 5a, the absorbance is reduced with the reduction in the PAN concentration and pH. An increase in the PAN concentration increases the absorbance in a medium amount of pH. The optimal absorbance is about 0.52 when the pH and concentration of PAN reach 9.73 and $6.6 \times 10^{-5} \text{ mol L}^{-1}$, respectively.

Figure 5b shows the interaction between extraction solvent volume and

concentration of PAN while pH kept at 9.73. According to the response surface plot, with increasing the extraction solvent volume to 300 μL , while concentration of PAN is $6.6 \times 10^{-5} \text{ mol L}^{-1}$, the absorbance is decreased. High absorbance is observed in the region when the extraction solvent volume and concentration of PAN are set at 152.27 μL and $6.6 \times 10^{-5} \text{ mol L}^{-1}$, respectively (Figure 5b). The absorbance is optimized when the pH and the extraction solvent volume are about 9.73 and 152.27 μL , respectively (Figures 5c). The counterplot of desirability function has shown optimal values of the variables (Figure 6).

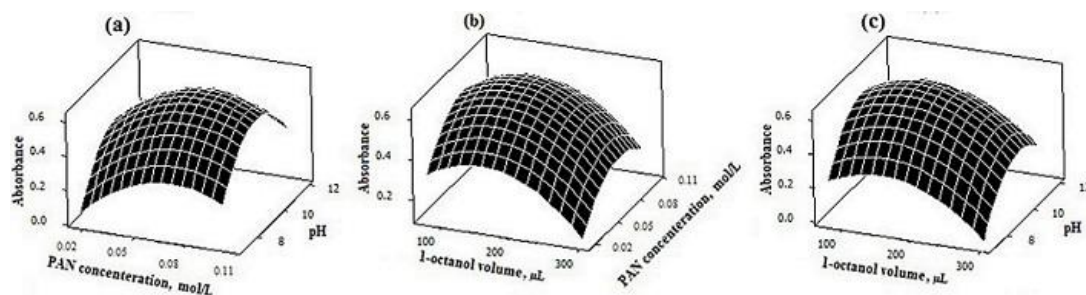


Figure 5. Response surface plots when optimizing the following pair of factors, while the other factor was kept constant at a central point (central level): (a) pH–concentration of PAN (extraction solvent, 152.27 μL); (b) concentration of PAN–volume of extraction solvent (pH, 9.73); (c) pH–volume of extraction solvent (concentration of PAN, 0.000066 mol L^{-1}).

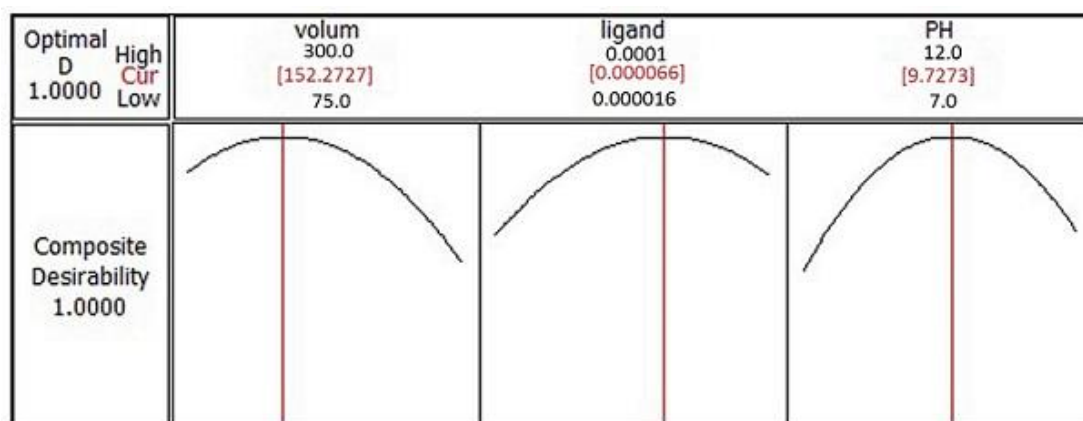


Figure 6. Desirability function

Interferences effects

Selectivity of the SS-DLPME method for determining nickel was assessed in the presence of various ions; therefore, effects of common coexisting ions on the micro-extraction of Ni (II) were investigated in optimum conditions. For this purpose, appropriate amounts of the other ions were added to 15 mL sample solutions containing 200 μgL^{-1}

of Ni^{2+} and the SS-DLPME procedure was performed. The results are given in Table 2. An ion was considered as interference when it caused a variation in the absorbance value of the sample more than 5%. The results showed that at these levels of concentrations, there aren't any significant interference.

Table 2. Effect of foreign ions on the recovery of 200 μgL^{-1} Ni (II) using SS-DLPME method

Ions	Ion / $[\text{Ni}^{2+}]$, μgL^{-1}
Hg^{2+} , Mn^{2+} , Cr^{3+}	25
Cd^{2+} , Ag^+ , Fe^{3+} , Pb^{2+} , Cu^{2+}	50
Zn^{2+} , Co^{2+}	100
Al^{3+}	1000
Mg^{2+} , K^+	2000
Cl^- , SO_4^{2-} , PO_4^{3-} , CH_3COO^-	1000

Validation and application of the SS-DLPME

The suggested SS-DLPME procedure was successfully applied for the determination of nickel in several water, vegetable and tobacco samples obtained in Arak and Malayer cities, Iran. To check the validity and accuracy of the developed method, each sample was spiked with the known amounts of Ni and the results along with the recovery were given in Table 3. The data showed that the relative Ni recoveries were between 95.9 and 104.33%. In addition, water certified reference material (TMDA 53.3, National Water Research Institute, Ontario, Canada) was analyzed by the proposed method for the pre-concentration and determination of the Ni ion. The obtained value was in good agreement with the certified value.

Analytical figures of merit

The analytical performance of the SS-DLPME method, including linear dynamic range (LDR), limit of detection (LOD), reproducibility and enrichment factor, were investigated under optimum conditions. A calibration curve of Ni (II) was linear

from 10 to 875 $\mu\text{g L}^{-1}$ with a correlation coefficient (R^2) of 0.9970. The limit of detection, LOD, evaluated as the ratio of three times standard deviation of eight blank signals to the slope of the calibration curve after pre-concentration, was 1.80 $\mu\text{g L}^{-1}$. To evaluate the precision of the method (RSD), the proposed method was tested and repeated seven times for 20 mL of the sample solution containing 200 $\mu\text{g L}^{-1}$ Ni which was 1.98% indicating good reproducibility of the method. Enrichment factor was obtained at about 86 which was calculated from the slope ratio of calibration curve with (1.4320) and without (0.0165) pre-concentration by the SS-DLPME procedure.

Comparison of SS-DLPME with other methods

Comparison of the main analytical characteristics including the values of LDR, RSD, EF/PF, LOD and sample volume of the SS-DLPME method with those reported in the literature for determination of nickel is given in Table 4.

Table 3. Determination of Ni in different real samples

sample	Certified (μgL^{-1})	Ni added (μgL^{-1})	Ni found ^a (μgL^{-1})	Recovery %
TMDA 53.3	311	-	306 ± 1.22	98.39 ± 0.67
		0	0.46 ± 1.21	-
Tap water (Malayer, Iran)		100	96.37 ± 1.70	96.37 ± 1.11
		200	201.79 ± 0.78	100.89 ± 0.39
		0	N.D ^b	-
Davijan river(Malayer, Iran)		100	102.89 ± 1.44	102.89 ± 1.24
		200	198.55 ± 1.76	99.27 ± 0.88
		0	1.45 ± 1.6	-
Well water(Eznavleh, Malayer)		100	97.06 ± 1.38	97.06 ± 1.08
		200	201.45 ± 1.52	100.72 ± 0.76

	0	N.D	-
Tap water (Arak, Iran)	100	99.68 ± 0.82	99.68 ± 0.42
	200	200.16 ± 0.96	100.08 ± 0.48
Mineral water	0	0.81 ± 0.89	-
	100	99.61 ± 1.19	99.61 ± 0.79
	200	200.80 ± 0.80	100.40 ± 0.40
Well water (Eznav, Malayer)	0	0.76 ± 0.71	-
	100	98.49 ± 1.85	98.49 ± 1.21
	200	200.76 ± 1.85	100.38 ± 0.92
Spinach	0	5.87 ± 0.20	-
	100	105.77 ± 0.32	99.90 ± 0.30
	200	203.40 ± 0.32	98.76 ± 0.16
Lettuce	0	4.84 ± 0.12	-
	100	105.13 ± 0.49	100.29 ± 0.39
	200	204.69 ± 0.66	99.92 ± 0.33
Tobacco	0	1.73 ± 0.12	-
	100	101.60 ± 0.49	99.87 ± 0.33
	200	200.40 ± 0.49	99.33 ± 0.24

^aMean value of three replicate determination ± standard deviation (n = 3). ^bNot. Detection.

Table 4. Comparison of the proposed method with other pre-concentration methods for nickel in analysis of different samples

Method	EF/PF	LOD($\mu\text{g L}^{-1}$)	LDR($\mu\text{g L}^{-1}$)	RSD%	Ref.
Co- precipitation/ FAAS	30	1.9	–	2.7	[19]
SPE/ FAAS	50	1.03	–	8	[20]
CPE/ FAAS	58	1.9	10 - 200	1.3	[21]
ISFME/ FAAS	153	0.6	2- 80	2	[22]
AA-HLLME / FAAS	340	3.6	10 - 450	2.1	[23]
IL-USE-ALLME/FAAS	21	7	7.0 - 667	9.1	[24]
CPE/FAAS	15.50	4.9	4.9 - 300	4.3	[25]
SS-DLPME/FAAS	86	1.80	10 - 875	2.05	this work

LOD: Limit of detection, LDR: linear dynamic range, RSD: Relative standard deviation, EF: enhancement factor, PF: pre-concentration factor.

According to Table 4, the proposed method has a low detection limit, wide linear dynamic range, good enhancement factor and low relative standard deviation, which are better, in most cases or comparable with several reported methods. In the proposed technique, low-density organic solvents are consumed instead of chlorinated solvents, which have a higher density

than water and less toxicity than chlorinated solvents. These results clearly reveal that the SS-DLPME is a reproducible, rapid, and simple technique that can be applied for pre-concentration and determination of trace amounts of nickel from water and vegetable and tobacco samples.

Conclusion

In this paper, a SS- DLPME method as a rapid and sensitive determination of Ni (II) ion was proposed. This method has some merits such as good enhancement factor, low detection limit, and great precision. In this study, the homemade extraction vessel was used in order to ease withdrawal of the extraction solvent in the sample solution. Plackett–Burman design (PBD) was applied for screening the various factors to specify significant factors which has an important role in the response. After screening, the significant factors were optimized using Box Behenken design (BBD). The model obtained from BBD is experimentally validated. Furthermore, it does not require any particular instruments (e.g. ultrasonic bath or vortex mixer), dispersive solvent, moreover the extraction is done in a close system; therefore, the probability of the organic solvent loss in the extraction process is minimized. The proposed method provided good repeatability, high EF, low LOD and a wide LR and could be used as a powerful technique for the pre-concentration and determination of Ni in various water, vegetable and tobacco samples.

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