

Scientific paper

Spectrophotometric Determination of Trace Levels of Nickel in Water Samples after Dispersive Liquid Liquid Microextraction Using 2,2 -Furildioxime as the Complexing Agent

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Abstract

A new method of dispersive liquid-liquid microextraction (DLLME) combined with UV-Vis spectrophotometry was proposed for the determination of trace nickel using 2,2 -Furildioxime as chelating and spectrophotometric reagent. In the proposed approach chloroform and ethanol were used as extraction and dispersive solvents, respectively. Some factors influencing the extraction efficiency of nickel and its subsequent determination were studied and optimized, such as the extraction and dispersive solvent type and volume, pH of the sample solution, extraction time and ionic strength. Under the optimal conditions, the calibration curve was linear within the range of 5–180 $\mu\text{g L}^{-1}$ of nickel with $R^2 = 0.9960$. Limit of detection ($3S_b/m$) was 0.6 $\mu\text{g L}^{-1}$ in the original solution and the relative standard deviation for ten replicate determination of 100 $\mu\text{g L}^{-1}$ nickel was 2.9%. The proposed method has been applied for the determination of nickel in various water samples with satisfactory results.

Keywords: Nickel, Dispersive liquid–liquid microextraction, Spectrophotometry, Water samples

1. Introduction

Nickel can be released into the atmosphere from several industrial processes, including oil and coal burning power plants, trash incinerators, and the metallurgy industry. Moreover, nickel can enter into aquatic bodies through the dissolution of oxide and sulfide rocks containing nickel combined with other elements. Thus, the exposure to nickel may take place by air, food samples, and drinking water, though mainly by skin contact with contaminated soils and alloys containing nickel.¹ Although nickel is considered an essential element, which acts as an activator of several enzymes to enhance insulin activity, excessive nickel in the organism is very toxic, which can be manifested by affecting the activity of natural killer cells.² In addition, chronic exposure to high nickel concentrations can cause cancer.³ Another common disease caused by skin disorder is known as nickel-eczema. This disease is more pronounced in workers

daily exposed to great amounts of nickel and in those people who are sensitive to nickel.⁴ Thus, because of its rising use, on the one hand, and its toxicity, on the other the determination of nickel is of great interest in environmental analysis.

The most common methods for the determination of nickel ions are flame and graphite furnace atomic absorption spectrometry,^{5–7} inductively coupled plasma optical emission spectrometry (ICP-OES)^{8,9} and spectrophotometry.^{10,11} Unfortunately, lower levels of the heavy metals like nickel than the quantitation limits of the instrumental techniques and higher levels of the concomitant ions in the real samples are two big and important problems for metal determinations in environmental samples. In order to solve these problems, separation and pre-concentration techniques are used for determining trace metal ions.^{12–14}

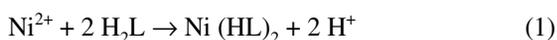
Some enrichment procedures have been developed for nickel determination involving different analytical

techniques such as liquid–liquid extraction,¹⁵ coprecipitation,¹⁶ cloud point extraction¹⁷ and solid-phase extraction.¹⁸ The applicability of some of these procedures is limited by certain disadvantages: they are time-consuming, sometimes provide unsatisfactory enrichment factors, use large volumes of organic solvents and produce secondary wastes. Cloud point extraction¹⁹ and liquid-phase microextraction²⁰ are fairly new methods of sample preparation, which have proved to be simple and inexpensive, opening up interesting ways to deal with the subject. However, it is necessary to establish an easy and rapid analytical method for quantitative determination of compounds.

Recently, a microextraction technique based on a ternary solvent system has been developed. In this technique, termed dispersive liquid–liquid microextraction (DLLME),²¹ an appropriate mixture of an extraction solvent and a dispersive solvent is rapidly injected into an aqueous sample so that a cloudy solution is formed. The analyte in the sample is then transferred to the fine droplets of the extraction solvent and phase separation is performed by centrifugation. The advantages of this method include its simplicity of operation, rapidity, low cost, low consumption of organic solvents and high enrichment factors. The technique has been applied to the determination of trace organic pollutants and metal ions in environmental samples.^{22–24}

Several techniques have been used for the determination of trace metals after a preconcentration step by DLLME such as flame atomic absorption spectrometry (FAAS),²⁵ electrothermal atomic absorption spectrometry (ET-AAS),²⁶ spectrophotometry²⁷ and inductively coupled plasma optical emission spectrometry (ICP-OES).²⁸ Among these techniques that are used in the determination of trace levels of metal ions, spectrophotometry continues to enjoy wide popularity. The common availability of the instrumentation, the simplicity of procedures, speed, precision and accuracy of the technique still make spectrophotometric methods attractive.

The aim of this work is to combine dispersive liquid–liquid microextraction (DLLME) with UV-Vis spectrophotometry to develop a new and simple procedure for the determination of trace nickel in various water samples. 2,2 -Furildioxime, used as chelating reagent to extract nickel ions into the organic phase. Ni (II) ion reacts with 2,2 -Furildioxime (H₂L) to form Ni(HL)₂ complex in aqueous medium²⁹:



2. Experimental

2. 1. Reagents and Solutions

All reagents used were of analytical grade. The stock standard solutions of nickel at a concentration of

500 mg L⁻¹ were prepared using Ni (NO₃)₂ 6H₂O. The working standard solutions were obtained by appropriate dilution of the stock standard solutions with doubly distilled water.

Other reagents used were: chelating agent 2,2 -Furildioxime (Fluka Chemie AG, Buchs, Switzerland), hydrochloric acid, nitric acid, sodium hydroxide, ethanol, methanol, acetone, acetonitrile, chloroform, dichloromethane, carbon tetrachloride and sodium nitrate were obtained from Merck (Darmstadt, Germany). Chlorobenzene was obtained from Fluka (Buchs, Switzerland).

The pipettes and vessels used for trace analysis were kept in 10% nitric acid for at least 24 hrs and subsequently washed four times with distilled water.

2. 2. Apparatus

A dual beam spectrophotometer model Cintra-20 from GBC (Australia) with 50 μL quartz micro-cells was used for conventional spectrophotometry. Phase separation was assisted using a centrifuge (Hettich, EBA 20). The pH-meter model 827 from Metrohm (Herisau, Switzerland) with combined glass electrode were used for the pH measurements.

2. 3. Extraction Procedure

To 5 mL of solution containing the analyte in a 10 mL test tube with conical bottom, 0.1 mL of 0.1 mol L⁻¹ ammonia buffer (pH = 9) and 0.1 mL of 0.01 mol L⁻¹ 2,2 -Furildioxime (chelating reagent) solution were added. By using a 1-mL syringe (gastight, Hamilton, Nevada, USA), 0.5 mL ethanol (dispersive solvent) containing 100 μL chloroform (extraction solvent) was added to the above solution. Chloroform was dispersed in all the parts of the sample and there was no need to homogenize the sample. In this step, the complex of Ni with 2,2 -Furildioxime was extracted into the fine droplets of chloroform. Then the mixture was immediately centrifuged for 5 min at 5000 rpm. The sedimented chloroform was removed using a 100 μL microsyringe and was made up to 200 μL by adding ethanol. The absorbance of solution was measured at the wavelength of maximum absorbance of complex, 433 nm.

3. Results and Discussion

In order to obtain high extraction efficiency, the effect of different parameters, influencing the complex formation and the extraction conditions, were optimized. These parameters were pH, the chelating agent concentration, the type and the volume of the extraction solvent, the type and the volume of the dispersive solvent, the extraction time and the ionic strength. Finally, these optimal conditions were applied to extract and detect nickel in the

real water samples. Triplicate extractions were performed for all experiments, and the average of these results was reported in figures or tables.

3. 1. Effect of pH

Because pH plays a unique role in the metal-chelate formation and the subsequent extraction, the pH of the sample solution was the critical factor evaluated for its effect on the DLLME preconcentration of Ni (II). In this work, 2,2 -Furildioxime was used as chelating reagent. A series of experiments were performed with the pH adjustment from 1 to 11 with hydrochloric acid and sodium hydroxide and the effect of pH on the extraction of nickel complex was investigated. The results are shown in Figure 1. As can be seen, the extraction recovery of nickel complex increases up to pH = 7, and then remains constant in the pH range 7–10. The competition between protons and nickel ions could explain the weak recovery in acid medium. This result is in agreement with results obtained by previous studies.^{30,31} Hence, ammonia buffer (pH 9.0) was used for pH adjustment in working solutions.

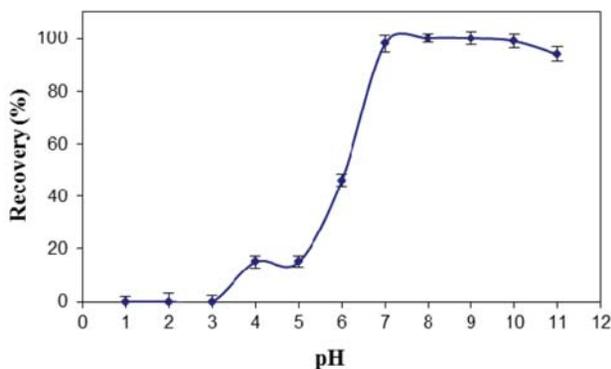


Figure 1. Effect of pH on the extraction recovery of nickel. Extraction conditions: water sample volume, 5.0 mL; 2,2 -Furildioxime concentration, 2×10^{-4} mol L⁻¹; extraction solvent (chloroform) volume, 100 μ L; dispersive solvent (ethanol) volume, 0.5 mL; concentration of nickel, 100 μ g L⁻¹.

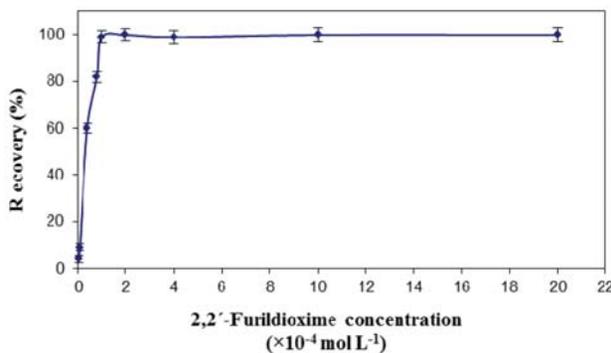


Figure 2. Effect of 2,2 -Furildioxime concentration on the extraction recovery of nickel. Extraction conditions: water sample volume, 5.0 mL; pH = 9; extraction solvent (chloroform) volume, 100 μ L; dispersive solvent (ethanol) volume, 0.5 mL; concentration of nickel, 100 μ g L⁻¹.

3. 2. Effect of 2,2 -Furildioxime Concentration

The influence of 2,2 -Furildioxime concentration as a chelating agent was studied in the range spanning from 4.0×10^{-6} – 2.0×10^{-3} mol L⁻¹. As is shown in Figure 2, by adding of an increased amount of 2,2 -Furildioxime, the extraction recovery of the target ion was increased before 1.0×10^{-4} mol L⁻¹ and then remained constant. At a low concentration of 2,2 -Furildioxime, the complexation was not complete and the extraction efficiency is low. Hence, 2.0×10^{-4} mol L⁻¹ 2,2 -Furildioxime was chosen to account for other extractable species.

3. 3. Selection of Extraction Solvent

The selection of an appropriate extraction solvent has great importance in the optimization of the DLLME method. The extracting solvent for DLLME must have low volatility, low toxicity, low water solubility and must not interfere with the analytical techniques used for the determination of analytes.²³ In the proposed method, the selected solvent should also have a density higher than water to sediment after centrifugation. According to these considerations, chlorobenzene (density, 1.11 g mL⁻¹), carbon tetrachloride (density, 1.59 g mL⁻¹), dichloromethane (density, 1.32 g mL⁻¹) and chloroform (density, 1.48 g mL⁻¹) were selected as the extraction phases and compared for the extraction of Ni(II)- 2,2 -Furildioxime complex from water. A series of sample solutions containing nickel were selected and DLLME procedure was followed by using 0.5 mL of ethanol containing 100 μ L of each extraction solvent. After DLLME sedimented phase was made up to 200 μ L by adding ethanol and the absorbance was measured. Figure 3 shows the effect of the type of extraction solvent on the extraction recovery of nickel. As can be seen, recovery with chloroform is quantitative. Therefore, chloroform was selected as the extraction solvent for further experiments.

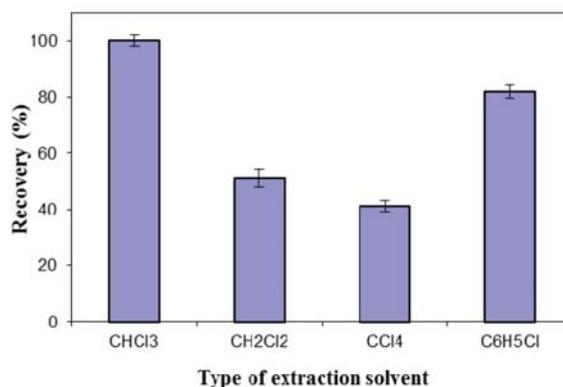


Figure 3. Effect of the type of the extraction solvent on the extraction recovery of nickel. Extraction conditions: water sample volume, 5.0 mL; pH = 9; 2,2 -Furildioxime concentration, 2×10^{-4} mol L⁻¹; dispersive solvent (ethanol) volume, 0.5 mL; extraction solvent volume, 100 μ L; concentration of nickel, 100 μ g L⁻¹.

3. 4. Effect of the Volume of Extraction Solvent

The effect of the volume of extraction solvent on the extraction recovery of nickel was investigated. Experiments were performed with different volumes of chloroform (in the range of 50 to 200 μL), as the extraction solvent, by fixing the volume of the ethanol at 0.5 mL. Figure 4 indicates that the extraction recovery increased by increasing the volume of chloroform to 100 μL and then remained constant. It is clear that at low volume of chloroform (lower than 100 μL) the amount of chloroform is not enough for a complete extraction of nickel; therefore extraction recovery is low. In the following studies, 100 μL was selected as the optimum volume of extraction solvent.

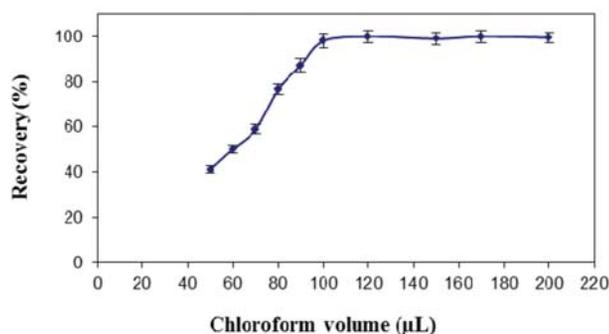


Figure 4. Effect of the volume of extraction solvent on the extraction recovery of nickel. Extraction conditions: water sample volume, 5.0 mL; pH = 9; 2,2 -Furildioxime concentration, 2×10^{-4} mol L^{-1} ; extraction solvent, chloroform; dispersive solvent (ethanol) volume, 0.5 mL; concentration of nickel, $100 \mu\text{g L}^{-1}$.

3. 5. Selection of Dispersive Solvent

The choice of a dispersive solvent was done considering the miscibility between organic phase (extraction solvent) and aqueous phase (sample solution). Thus, acetonitrile, acetone, ethanol and methanol were particularly

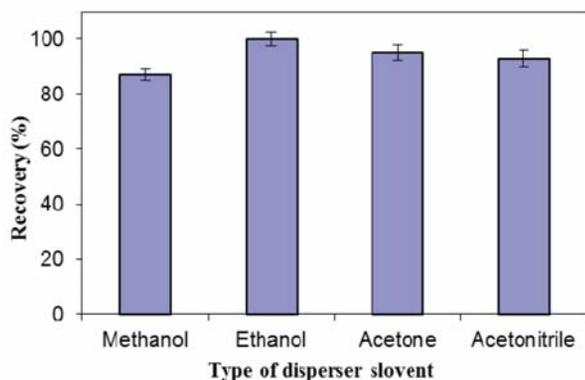


Figure 5. Effect of the type of the dispersive solvent on the extraction recovery of nickel. Extraction conditions: water sample volume, 5.0 mL; pH = 9; 2,2 -Furildioxime concentration, 2×10^{-4} mol L^{-1} ; extraction solvent (chloroform) volume, 100 μL ; dispersive solvent volume, 0.5 mL; concentration of nickel, $100 \mu\text{g L}^{-1}$.

evaluated. Recovery efficiency was evaluated using 0.5 mL of each dispersive solvent containing 100 μL extraction solvent (chloroform). Ethanol yielded the highest recovery for Ni (Figure 5), and thereby this solvent was selected as the dispersive solvent for our studies. This higher recovery can be attributed to the better dispersion obtained with ethanol.

3. 6. Effect of the Volume of Dispersive Solvent

The volume of dispersive solvent directly affects extraction solvent solubility in aqueous phase and thus influencing the efficiency of the microextraction technique. Thus, ethanol volumes ranging within 0.25–2 mL were assayed. As can be seen in Figure 6, the extraction recovery reached to its maximum value at 0.5 mL of the ethanol and then remained approximately constant by further increasing its volume from 0.5–1 mL. At higher volumes of ethanol, the recovery of the nickel decreases, because of increasing the solubility of the complex in the aqueous samples. Therefore, 0.5 mL of ethanol was selected as the optimum volume of dispersive solvent.

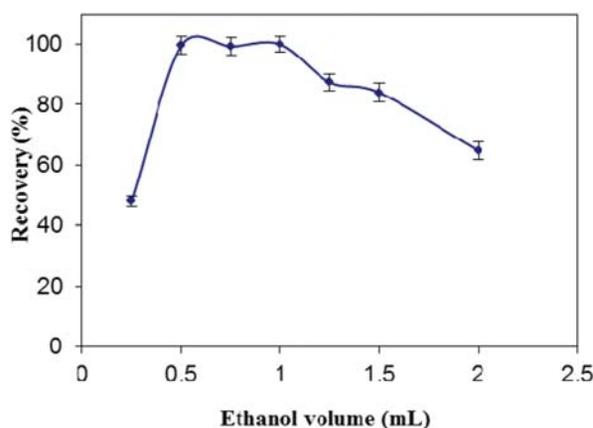


Figure 6. Effect of the volume of dispersive solvent on the extraction recovery of nickel. Extraction conditions: water sample volume, 5.0 mL; pH = 9; 2,2 -Furildioxime concentration, 2×10^{-4} mol L^{-1} ; extraction solvent (chloroform) volume, 100 μL ; dispersive solvent, ethanol; concentration of nickel, $100 \mu\text{g L}^{-1}$.

3. 7. Effect of Extraction Time

The effect of extraction time (interval time between the injection of a mixture of dispersive solvent and extraction solvent, before starting to centrifuge) on the performance of DLLME is considered as a key factor which must be studied and evaluated. Therefore, for evaluating this parameter, different extraction times (ranging from 0 to 30 min) with constant experimental conditions were studied. The results clearly revealed that the proposed extraction method is very fast so that the extraction time has no measurable effect on the extraction efficiency. This is

mainly due to an infinitely large surface area between extraction solvent and aqueous phase. Such a short extraction time can be considered as one of the main advantages of the DLLME method, as reported before.²³

3. 8. Effect of Centrifugation Rate

To achieve a good separation result, the effect of centrifugation rate on the extraction recovery were studied in the range of 1000–6000 rpm. It was found that over 4000 rpm, organic phase completely settled, so the rate of 5000 rpm was selected as the optimum point.

3. 9. Effect of Centrifugation Time

Centrifugation is an important procedure for separating extraction solvent from aqueous solution in the proposed method, and centrifugation time could affect the volume of sedimented phase. In order to attain the best extraction efficiency, the centrifugation time was optimized with the time span from 1 to 30 min at a rotation speed of 5000 rpm. According to the results, a centrifugation time of 5 min was selected as optimum, since complete separation occurred during this time and no appreciable improvements were observed for a longer interval.

3. 10. Influence of Ionic Strength

In order to investigate the influence of the ionic strength on the DLLME performance, several experiments were performed with different NaNO₃ concentrations (0.0–1.0 mol L⁻¹) while keeping other experimental parameters constant. According to the obtained experimental results, salt addition has no significant effect on extraction recovery. Therefore, all the extraction experiments were carried out without adding salt.

3. 11. Interference Effects

The influence of several ions on the preconcentration and determination of nickel was examined using 100 µg L⁻¹ nickel standard solution in the presence of various amounts of individual ions. An ion was considered to in-

Table 1: Tolerance limits of coexisting ions on the DLLME of Nickel.

Interfering ions	Ion/Ni (II) ratio
K ⁺ , Na ⁺ , Li ⁺ , Ca ²⁺ , Mg ²⁺ , F ⁻ , I ⁻ , NO ₃ ⁻ , PO ₄ ³⁻	1000
Mn ²⁺ , Cd ²⁺ , CrO ₄ ²⁻ , Au ³⁺ , ReO ₄ ⁻	100
Br ⁻ , Pd ²⁺	50
Ba ²⁺ , Pb ²⁺	30
Fe ³⁺ , Zn ²⁺	25
Cr ³⁺ , Ag ⁺	15
Fe ²⁺ , UO ₂ ²⁺	10
Cu ²⁺	3
Al ³⁺ , Co ²⁺	2

terfere when its presence produced a variation on the recovery higher than ± 5%. The results are shown in Table 1 and prove that Ni recoveries are almost quantitative in the presence of interfering ions.

3. 12. Analytical Performance

Table 2 shows the analytical characteristics of the method. Under the optimum experimental conditions, the calibration curve for nickel was linear from 5 to 180 µg L⁻¹. The limit of detection (LOD), defined as LOD = 3S_B/m (where LOD, S_B, and m are the limit of detection, standard deviation of the blank and slope of the calibration graph, respectively), was 0.6 µg L⁻¹. The preconcentration factor of 25 was obtained with a sample volume of 5 mL.

Table 2: Analytical Characteristics of the method.

Analytical feature	Parameter
Linear range (µg L ⁻¹)	5–180
Limit of detection, µg L ⁻¹ (n = 10)	0.6
RSD ^a (%)	2.9–3.2
Preconcentration factor	25
Extraction recovery ^b (%)	> 98

^a Ni (II) concentration, 10, 100 and 150 µg L⁻¹

^b Ni(II) concentration 10, 100 and 150 µg L⁻¹

3. 13. Analysis of Real Samples

The proposed method was applied to the extraction and determination of nickel in different water samples (tap, mineral, spring and sea water samples) and the results are summarized in Table 3. According to the results,

Table 3: Determination of nickel in water samples.

Recovery	Ni(II) detected (%)	Ni(II) added (µg L ⁻¹)	Sample (µg L ⁻¹)
Tap water ^a	0.0	n.d ^e	–
	50.0	49.5 (3.2) ^f	99.0
	100.0	98.0 (3.1)	98.0
Mineral water ^b	0.0	n.d ^e	–
	50.0	48.0 (3.3)	96.0
	100.0	97.0 (3.5)	97.0
Spring water ^c	0.0	n.d ^e	–
	50.0	47.5 (3.2)	95.0
	100.0	96.0 (3.3)	96.0
Sea water ^d	0.0	n.d ^e	–
	50.0	51.0 (3.5)	102.0
	100.0	101.0 (3.6)	101.0

^a From drinking water system of Sari, Iran, ^b From Damavand mineral water, Damavand, Iran, ^c From Arteh village, Sari, Iran, ^d Caspian sea water, Babolsar, Iran, ^e Not detected, ^f RSD of three replicate experiments

the concentration of nickel in the analyzed water samples was below the LOD of the method. The suitability of the proposed method for the analysis of natural water samples was checked by spiking samples with 50 and 100 $\mu\text{g L}^{-1}$ of nickel. The relative recoveries of the method (expressed as the mean percentage between the amounts determined and the ones spiked) for the water samples were in the range spanning from 95.0 to 102.0% with the relative standard deviations (RSDs) of 3.1–3.6%. These results indicate a good performance of this method for the determination of Ni in various water samples.

3. 14. Comparison With the Other Methods

A comparison of the presented method with the other reported preconcentration methods for the nickel extraction and determination from water samples is given in Table 4. In comparison with other reported methods, the proposed method has low LOD and good preconcentration factor. The method developed in this work is proposed as a suitable alternative to more expensive instruments for Ni determination at trace levels. This methodology is a reproducible, simple and low cost technique and does not require further instrumentation. These characteristics are of great interest for the routine laboratories in the trace analysis of metal ions.

4. Conclusions

The main difficulty in the determination of nickel in natural waters is its low concentration level. This study demonstrates the successful application of the DLLME method whereby separation and preconcentration of ng

mL^{-1} levels of nickel in several water samples can be achieved. Simplicity of operation, sensitivity, rapidity, minimum use of toxic organic solvent and selectivity are the advantages of the present method. The results showed that this method provides high recovery and good preconcentration factor within a short time and good linearity over the investigated concentration range. In this method, sample preparation time as well as consumption of toxic organic solvents was minimized without affecting the sensitivity of the method. This is a novel method and is suitable for simple and accurate determination of this element in a variety of water samples with satisfactory results.

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Table 4: Comparison of DLLME with other extraction methods for determination of nickel in water samples.

Method	LOD ^a ($\mu\text{g L}^{-1}$)	R.S.D. ^b (%)	PF ^c	Sample volume (mL)	Reference
FI–coprecipitation–FAAS	1.4	2.2	22 ^d	50	32
On line–SPE–FAAS	4.1	6.3	20.9	20	33
CPE–FAAS	2.7	1.8	17	25	34
CPE–ICP–OES	6.3	2.6	9.79	15	8
CPE–spectrophotometry	10	3.6	10	10	35
SPE–FAAS	0.75	0.9	160	800	36
MF–FAAS	7.8	<10	60	300	37
Coprecipitation–FAAS	3	–	100	200	38
Flotation–FAAS	0.7	<1	93	750	39
BE–FAAS	12.5	3.1	40	10	40
Coprecipitation–FAAS	1.41	–	120	600	41
Coprecipitation–FAAS	1.9	2.7	30	150	42
DLLME–FAAS	1.4	1.5–5.5	29 ^d	10	43
DLLME–ICP	0.4	3.8	8	10	44
DLLME–Spectrophotometry	0.6	2.9	25	5	Present work

FI; flow injection, FAAS; flame atomic absorption spectrometry, SPE; solid phase extraction, CPE; cloud point extraction; OES; optical emission spectroscopy, MF; membrane filtration, BE; back extraction, ^a Limit of detection, ^b Relative standard deviation, ^c Preconcentration factor, ^d Enhancement factor

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Povzetek

Za določanje sledov niklja predlagamo novo metodo z disperzivno mikroekstrakcijo tekoče-tekoče (DLLME) v kombinaciji z UV-Vis spektrofotometrijo z 2,2'-furildioksimom kot kompleksantom in spektrofotometričnim reagentom. Pri predlaganem pristopu smo kot ekstrakcijsko in dispergirno topilo uporabili kloroform ter etanol. Proučevali in optimizirali smo nekatere dejavnike, ki vplivajo na ekstrakcijsko učinkovitost za nikelj ter posledično na njegovo določitev, na primer vrsto in prostornino ekstrakcijskega in dispergirnega topila, pH raztopine vzorca, čas ekstrakcije, ionsko moč. Pod optimalnimi pogoji je bila umeritvena krivulja linearna v območju 5–180 g L⁻¹ niklja z R² = 0,9960. Meja zaznave (3S_b/m) je bila 0,6 g L⁻¹ v začetni raztopini, relativni standardni odklik za deset ponovitev določitev pri 100 µg L⁻¹ niklja pa 2,9%. Predlagano metodo smo z zadovoljivimi rezultati uporabili za določitev niklja v različnih vodnih vzorcih.