Spectrophotometric Determination of Nalidixic Acid in Some Real Samples After Preconcentration with GO/ZnO Nanocomposite-Based Pipette Tip Micro-Solid Phase Microextraction

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Abstract

In this research a graphene oxide/zinc oxide nanocomposite (GO/ZnO) was synthesized and employed for simple and sensitive pipette tip-based micro-solid phase extraction (PT-µSPE) of nalidixic acid (NA) from seawater and human blood plasma samples following by its determination by spectrophotometry. Several parameters affecting the suggested protocol were optimized, namely type and volume of eluent, amount of sorbent, sample volume, number of cycles of extraction and elution, pH of sample solution and type and amount of salt. The optimization performed by both response surface methodology (RSM) and one-variable-at-a-time techniques. Figures of merit were achieved as: detection limit; 0.30 µg L^{-1}, enrichment factor 40 for NA, linear calibration curve in the range of 1.0-200.0 µg L^{-1}; and reproducibility (as RSD %) better than 4.4%. Results for the application of the technique in seawater and human blood plasma showed that this analysis method can be applied for the determination of the analyte in complex real samples successfully.

1. INTRODUCTION

Nalidixic acid (NA) as quinolone derivative, that was the first 1.8-naftridine analyte synthesized with Lesher et al., is utilized in treatment of infectious diseases. This acid acted on the sensitive microorganisms in the multiplication phase, indicating the replication of the bacterial deoxyribonucleic acid, but without any effect on the nuclear synthesis of the eucariotic cells. NA is generally used against infections of the urinary tract, due to it is readily absorbed by the oral tract. Besides prescriptions for human, NA is also used in fish factories, as food additive or directly in aquatic media to avoid epidemic diseases [1-3].

As a result, determination of this compound in real samples such as seawater is of importance. Accordingly, monitoring and determination of trace level nalidixic acid in different real samples is still a vital task. Different analytical techniques have been utilized for determination of this analyte in various real samples, including spectrophotometry [1], high performance liquid chromatography [1], flourometry [1], voltammetry [3] and phosphorimetry [4]. Because in real samples with complicated media such as seawater, and due to necessitate of detecting low concentration of these analytes, their direct determination by analytical instruments is difficult and preliminary pretreatment and

preconcentration procedure is often requires [5]. Hereupon, different sample preparation approaches such as solid phase extraction (SPE) [6,7], solid phase microextraction [7], magnetic metal-organic framework nanocomposite [7], ultrasound-assisted ionic liquid dispersive liquid–liquid microextraction [8] and dispersive liquid microextraction [9] have been applied for this propose. Among these techniques, SPE is a favor method owing to its ease of operation, easiness automation and low cost [10]. However, while oppositely drawbacks like large amount of adsorbent and huge volume of organic solvents consumption restricted more and frequent application of SPE which encourage researches to design a miniaturized SPE technique namely pipette tip micro solid phase extraction (PT-µSPE). The technique is being quite efficient for extraction and/or preconcentration of target analytes, especially in complex media. The promising protocol is less expensive, easier to operate and providing excellent extraction efficiency, high reproducibility and versatility [11-13].

Graphene a single layer of carbon atoms densely packed into a benzene ring structure was introduced with Novoselov et al. in 2004 that utilized as a sorbent for SPE at the first time for the extraction of eight chlorophenols. Because of

its chemical stability, high sorption capacity, excellent thermal stability, strong hydrophobicity, high specific area and large delocalized π-electron system, the adsorbent was subsequently utilized for the enrichment and extraction of various analytes [14].

Graphene oxide (GO, a precursor of chemically converted graphene) include many oxygen atoms in the basal plane and edge of its sheets in the form of epoxy, hydroxyl and carboxyl groups [14].

Zinc oxide (ZnO) semiconductor, having various morphologies, and so have different favorable properties; including good transparency, high electron mobility, wide band gap and room-temperature luminescence. Consequently, loading ZnO nanoparticles on graphene oxide sheets can provide a higher available surface area and enhancement in adsorption capacity that has been applied for preconcentration of analyte such as nalidixic acid, recently [14].

Optimization protocols are usually performed by univariate technique which means one factor at a time. Besides being time consuming and laboring, the method do not involve the interaction between factors. So, design of experiment technique that is based on multivariate static methods is suitable replacement for one factor at a time techniques to investigate and optimize the effective factors on protocols. They are more precise in estimating effect of each parameter and interaction between parameters. One of the efficient statistical methods that are utilized excessively in optimization protocols is response surface methodology (RSM) [15-17].

RSM is a powerful multivariate method for designing experiments, building models and analyzing effects of various factors on each other to maximize response of criteria that is utilized. The advantages of its application on optimization of analytical protocols are generally disseminated, since the design of polynomial experiments allows simultaneous testing of different factors with potential influence on the response, producing large amounts of information from a relatively small number of runs and as a result, saves time, energy and chemicals [15-17].

The aim of this research is to develop a simple, rapid, sensitive and accurate protocol for the spectrophotometric determination of nalidixic acid in seawater samples and human blood plasma. The other objective is to combine the excellent adsorption performance of graphene oxide/zinc oxide nanocomposite (GO/ZnO), and simplicity, low solvent and sample consumption, and rapidity of pipette tip micro solid phase extraction (PT μSPE) for determination of the analyte. We call this technique as graphene oxide/zinc oxide nanocomposite pipette tip micro solid phase extraction (GO/ZnO PT μSPE). The parameters for sample pretreatment and analytical performance of PT μSPE- spectrophotometer technique are optimized using both response surface methodology (RSM) and one-variable-at-a-time.

2. EXPERIMENTAL

2.1. Materials and methods
Nalidixic acid was provided by Sigma-Aldrich Co. (St. Louis, MO, USA). Graphite powder (particles size <50 μm, 99.5% purity), potassium permanganate, sodium nitrate ($NaNO_3$), zinc sulphate ($ZnSO_4$), hydrazine hydroxide (99%) and H_2O_2 (30%) were obtained from Fluka AG (Switzerland). All salts, organic solvent and acids were of analytical grade and were purchased from Merck KGaA (Darmstadt, Germany). Milli-Q® water (18.3 MΩ/cm) was applied throughout the runs. Stock solutions of nalidixic acid were prepared at 500 mg L^{-1} in deionized water and stored at 4 ^{0}C in dark. Working solutions were diluted at various concentrations with Milli-Q water daily.

2.2. Apparatus
The analysis of solution and samples were carried out with a photonix Ar model 2015 UV-Vis spectrophotometer (Iran) equipped by two 100 μL quartz microcells (model Q-01701, Stara company), at the wavelength of 320 nm for nalidixic acid. The pHs of the solutions were determined using a 630 Metrohm (Switzerland) pH meter. Fig. 1 depicts the absorption spectra of NA at 175 μg L^{-1} concentration.
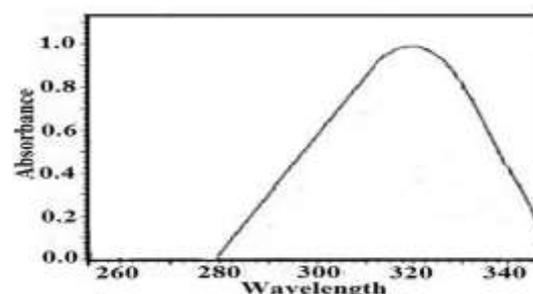

Fig. 1. Absorption spectra for 175 μg L^{-1} of nalidixic acid after GO/ZnO PT-μSPE.

2.3. Preparation and purification
Graphite oxide (GO) was synthesized from graphite with a modified Hummers protocol [14]. Briefly, 1.0 g of graphite, 6.0 g $KMnO_4$ and 1.0 g $NaNO_3$ were mixed in 46 mL of 98% sulphuric acid. Next, the mixture was stirred for 3 h in an ice bath. After that, 100 mL of deionized water was added gradually, and temperature was maintained at 90 ^{0}C for 30 min while the solution was stirred constantly. Then, 300 mL of deionized water was added slowly, and temperature was reduced to 40 ^{0}C. After that, 10 mL of hydrogen peroxide (30%) was added dropwise and the reaction solution was centrifuged. The solid product was separated via centrifugation and washed using 0.1 M hydrochloric acid and then deionized water until the pH of the washing water reached 7. The achieved solid was dried at 60 ^{0}C for 8 h in an oven. A schematic of the preparation of graphite oxide is shown in Fig. 2.
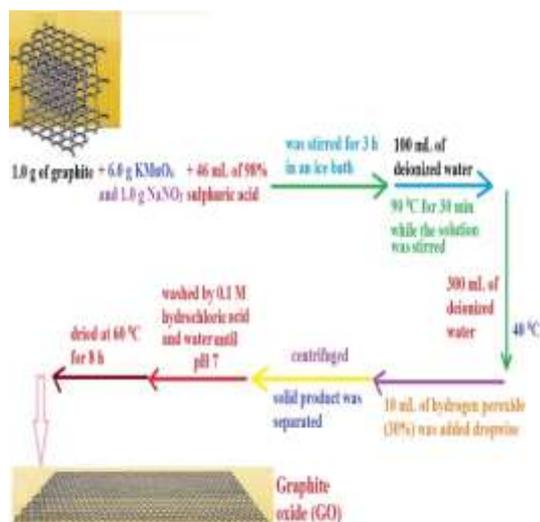
Fig. 2. Schematic of the preparation of graphite oxide.

2.4. Preparation of zinc oxide (ZnO)

For the synthesis of ZnO, 2.0 g $ZnSO_4$ was dissolved in NaOH solution and stirred for 12 h. The obtained white product was washed several times using distilled water and was collected with filtration. Finally, it was dried in air to achieve zinc nano oxide [14].

2.5. Preparation of GO/ZnO nanocomposite

For preparation of GO/ZnO hybrid, 5 g graphene oxide was dissolved in 20 mL ethanol and kept under ultrasonication for 2 h that was denoted as GO solution. 5 g of the as-prepared ZnO nanoparticles was poured into a solution including 18 mL methanol and 2 mL chloroform and kept under ultrasonication for 2 h which was denoted as ZnO solution. Then the GO and the ZnO solutions were mixed together and kept under constant stirring for 24 h. The precipitate was collected by centrifugation and washed by methanol several times. The final product was freeze-dried overnight and named GO/ZnO nanocomposite [14]. Characterization of prepared ZnO and GO/ZnO nanocomposite was carried out and presented in one of our previous works [14].

2.6. Procedure of GO/ZnO PT-µSPE

GO/ZnO as sorbent material (2.5 mg) was packaged into a 150 µL volume pipette-tip, and tip was attached to a commercial 10 mL syringe. Before performing extraction, GO/ZnO was activated by passing 1 mL of methanol following by 1 mL of ultra-purified water through it. A 10 mL aliquot of sample was taken and adjusted to pH 3.0 and 300 mg of Na_2SO_4 was added to it. pH of the solutions were adjusted by dropwise addition of either 1 mol L^{-1} of HCl or 1 mol L^{-1} NaOH. The analyte was loaded on sorbent with passing the solution for 7 times. Each time, 0.5 mL of the sample was taken into the syringe. Next, the analyte was eluted by 9 portions of 250 µL of elusion solvent. Pure methanol was used for this purpose. Finally, the eluent was transferred to a micro-cell for concentration determination of nalidixic acid by spectrophotometry. Schematic of the GO/ZnO PT-µSPE extraction is shown in Fig. 3.
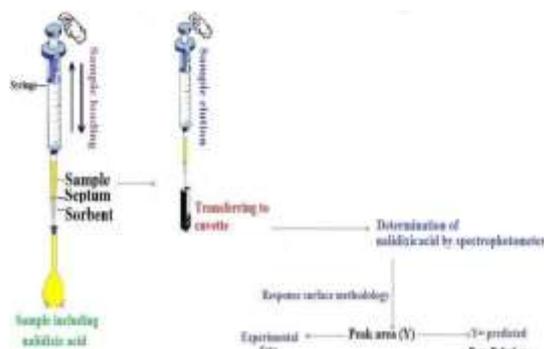

Fig. 3. Schematic of the GO/ZnO PT-µSPE extraction procedure.

3. RESULTS AND DISCUSSION

3.1. Optimization of GO/ZnO PT-µSPE

To optimize GO/ZnO PT-µSPE for the extraction of NA, some factors which significantly effect on the extraction efficiency have been investigated using both response surface methodology (RSM) and one-variable-at-a-time. Each run was carried out in triplicate (n=3) and a standard solution of 150 µg L^{-1} was analyzed. Parameters studied were type and volume of eluent, amount of material, number of cycles of extraction and elution, pH and volume of sample.

3.2.1. Eluent type

The choice of suitable solvent for elution is one of the most important steps in the elution of the analyte adsorbed on GO/ZnO. To elute the analyte efficiently and with the minimum amount of solvent, various organic solvents including methanol, acetonitrile, ethanol, water, methanol/acetic acid (with 1:2, 1:1 and 2:1 ratio), methanol:water (1:1), methanol contain 5% acetic acid, acetone, HCl (0.5 and 1.0 mol L^{-1}) and acetic acid were investigated. Methanol for NA has been chosen because they used higher absorption signals were achieved for the analyte. This is may be due to the fact that these eluents are polar aprotic solvents with relatively high dielectric constants and hence can readily dissolve NA.

3.2.2. Effect of amounts of GO/ZnO

Various amounts, i.e. 1.0 to 4.0 mg of GO/ZnO as sorbent, were evaluated in adsorption process. It was observed that the quantity of 2.5 mg can better do recovery of the analyte. By further increase in GO/ZnO loading, the recovery of the analyte was decreased, probably because sample passage and elution of pipette tip with higher amounts of sorbent become more difficult.

3.2.3. Effect of sample volume

Effect of sample volume on extraction efficiency was evaluated by increasing the sample volume from 2.0 mL to 12.0 mL while the concentration of analyte was kept fixed at 100 µL. Recoveries were improved continuously by increasing the sample volume up to 10 mL due to increasing the number of moles of the analyte available for extraction. Further increase in sample volume causes reduction in recoveries, due to dilution of the analyte. As a result, 10 mL sample

volume was selected to investigate the next experiments, since demonstrated the best recovery (>97%).

3.2.4. Effect of pH

The effects of the pH value on extraction efficiency of the analyte from aquatic samples were investigated in the range of 2.0-12.0. The pH of the solution was adjusted to the required value and extraction procedure was performed. The extraction efficiency was at highest point at the pH of 3.0. The value was selected as optimum pH of extraction.

3.1.5. Effect of type of salt

The salting-out effect is generally utilized in traditional liquid-liquid extraction to reduce solubility of target compounds in aqueous phase and consequently causes more analytes can enter into the extracting phase. Here we tried to find the effect of type of salt on extraction efficiency of NA by GO/ZnO PT-µSPE. KCl, NaCl and Na_2SO_4 were selected for this purpose. Our studies showed that Na_2SO_4 is the best reagent for salting out of NA and can increase recovery more than the other selected salts.

3.2.6. Response surface methodology

An optimization process was carried out to choose the best experimental condition for micro solid phase extraction of NA by RSM. RSM is a type of experimental design techniques that utilize a sequence of designed experiments to achieve an optimal response [15-17]. In this study, significant variable are volume of sample solution (A or X_1), number of cycles of extraction (B or X_2), number of cycles of elution (C or X_3) and amount of salt (mg) (D or X_4). The low, middle and high levels of each parameter were designated as -1, 0, +1, respectively (Table 1). The actual design of runs is shown in Table 2.

In a system such as four significant variables, X_1 (A), X_2 (B), X_3 (C) and X_4 (D), the mathematical relationship of the response on these variables can be approximated utilizing quadratic (second degree) polynomial equation (equation 1):

$$Y=\beta_0+\sum\beta_iX_i+\sum\beta_{ii}X_{ii}+\sum\beta_{ij}X_iX_j+e \qquad (Eq.\ 1)$$

Where Y is predicted response, β_0 is the constant, X_1, X_2, X_3 and X_4 are the coded independent variables, β_i is the linear effect, β_{ii} is the quadratic effect, β_{ij} demonstrates the coefficient of the interaction factor, and e is the random error or allows for description or uncertainties between predicated and determined values [15-17]. In equation 2, the mathematical relationship of the response and four variables is indicated.

Sqrt (Y, for NA)= Sqrt (Absorption) = -7.78983 + $(0.037435 \times A) + (0.31870 \times B) + (0.42384 \times C) + (6.12038 \times 10^{-3} \times D) - (4.85201 \times 10^{-5} \times A \times B) - (2.09558 \times 10^{-5} \times A \times C) - (1.32379 \times 10^{-3} \times B \times C) + (2.05305 \times 10^{-5} \times B \times D) - (2.51201 \times 10^{-5} \times C \times D) - (7.55372 \times 10^{-5} \times A^2) - (0.020335 \times B^2) - (0.021179 \times C^2) - (9.61982 \times 10^{-6} \times D^2)$ (Eq. 2)

The critical point in the surface can be obtained by solving these equation systems for the condition of $\partial(Y)/\partial(A) =0$, $\partial(Y)/\partial(B) =0$, $\partial(Y)/\partial(C) =0$ and $\partial(Y)/\partial(D) =0$ [43-45]. The summary of analysis of variance (ANOVA) is explained in Table 3. The obtained values for critical point are as follows: volume of eluent solvent (A) = 244, number of cycles of

extraction (B)= 7.4, number of cycles of elution (C)= 9.5 and amount of salt= 314.

Table 1. The levels of variables chosen for the trials.

Analyte	A	B	C	D
	200 (-1)	5 (-1)	7 (-1)	250 (-1)
NA	250 (0)	7 (0)	9 (0)	300 (0)
	300 (+1)	9 (+1)	11 (+1)	350 (+1)

Table 2. Response surface methodology design for the optimization of GO/ZnO PT-µSPE.

A	B	C	D	Y (NA)observed	Y (NA)predicted	Error (NA)
-1	-1	-1	-1	0.211	0.227	-7.51
-1	-1	-1	+1	0.243	0.254	-4.46
-1	-1	+1	-1	0.352	0.335	4.96
-1	-1	+1	+1	0.368	0.355	3.51
-1	0	0	0	0.555	0.586	-5.67
-1	+1	-1	-1	0.324	0.310	4.25
-1	+1	-1	+1	0.361	0.351	2.68
-1	+1	+1	-1	0.404	0.407	-0.72
-1	+1	+1	+1	0.447	0.440	1.49
0	-1	0	0	0.651	0.637	2.19
0	0	-1	0	0.648	0.617	4.74
0	0	0	-1	0.754	0.764	-1.36
0	0	0	0	0.875	0.831	5.04
0	0	0	0	0.883	0.831	5.90
0	0	0	0	0.880	0.831	5.58
0	0	0	0	0.885	0.831	6.11
0	0	0	0	0.874	0.831	4.93
0	0	0	0	0.888	0.831	6.43
0	0	0	+1	0.764	0.811	-6.21
0	0	+1	0	0.696	0.753	-8.25
0	+1	0	0	0.674	0.744	-10.36
+1	-1	-1	-1	0.165	0.163	1.08
+1	-1	-1	+1	0.189	0.186	1.47
+1	-1	+1	-1	0.238	0.248	-4.09
+1	-1	+1	+1	0.263	0.265	-0.93
+1	0	0	0	0.446	0.462	-3.56
+1	+1	-1	-1	0.205	0.216	-5.60
+1	+1	-1	+1	0.246	0.251	-2.04
+1	+1	+1	-1	0.309	0.289	6.39
+1	+1	+1	+1	0.338	0.318	6.05

The regression model for the analytes resulted in a determination coefficient ($R^2= 0.9879$, showing that only 1.21% of the variation cannot be described by the model. The adjusted determination coefficient at adjusted $R^2= 0.9780$ confirmed that the model was highly significant. Additionally, the prediction R^2 of 0.9584 was in acceptable agreement with R^2 and indicated a high predictive power of model. The results of analysis of variance (ANOVA) are presented in Table 3. The model *F*-value implies the model is significant. A *p*-value lower than 0.0001 was found, demonstrating again the high significance of the regression models. A *p*-value less than 0.05 in the ANOVA table explains the statistical significance of an effect at 95 % confidence level. The values in Table 3 show that the factors A, B, C and D, their interactions and quadratic terms NA is significant model terms. Significant models terms probably have a real effect on the absorbance (response).

As shown in Table 3, the *F*-value of Lack of Fit (LOF) of 2.52 expressed that the LOF was not significant relative to the pure errors. The small difference

between the values predicted with this model and the experimental result, the variance analysis of the model and the insignificant lack of fit, all explain that the accuracy and the fitness of the model were highly satisfactory. A high degree of precision and a good deal of the reliability of the coefficient of variation (CV= 3.91) was obtained. The ANOVA of the regression model showed that the quadratic model was significant, as was evident by the Fisher'S F-test (F_{model}= 100.28) by a very low probability value (p) ($p_{model} \leq 0.0001$). In Fig. 4, the correlation between the observed and predicted absorbance values of NA are showed. The points cluster near the diagonal line indicates a good fit of the model, that's because the derivation between the obtained and predicted values is small. Fig. 5 shows two dimensional response surfaces as the functions of two variables at the center level of other variables.
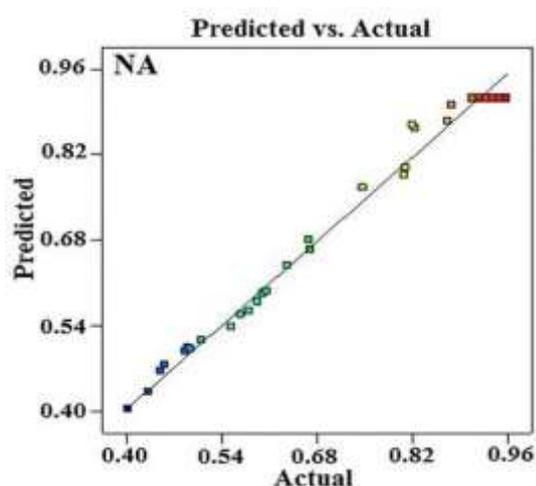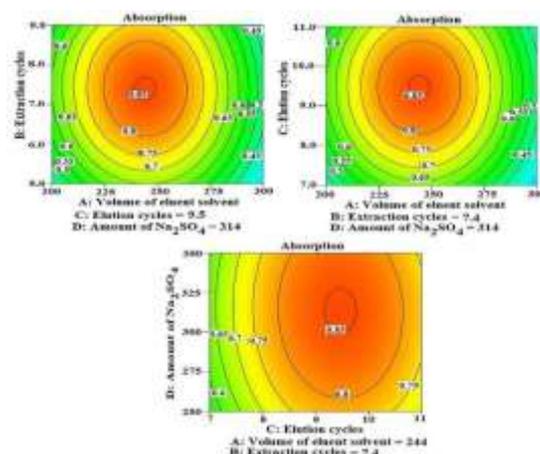

Fig. 5. Response surface -2D contours explaining the effect of independent variable on absorbance.

3.3. Analytical performance
3.3.1. Linear range, limit of detection and enrichment factor

The analytical validity of proposed GO/ZnO PT-μSPE method for the extraction and spectrophotometric determination of NA was verified by investigating linearity of its calibration range and their limit of detection. Under the optimized conditions, the linear range of 1-200 μg L^{-1} for the analyte was achieved. The least square equation over the dynamic range was:
A (for NA) = 0.0054 C (μg L^{-1})+0.052 with R^2= 0.9961
Where C and A are the concentration of the analyte and their absorbance, respectively. The percent extraction (E%) of the analyte was achieved using equation (3).
E%=100(C_B/C_A)   (Eq. 3)
The LOD of 0.30 μg L^{-1} for NA was obtained, based on 3S_d calculation [18]. Where S_d is the standard deviation of 10 measurement of their corresponding blanks.

Fig. 4. Parity plot show the correlation between the achieved and predicted value.

<div style="text-align:center">Table 3. ANOVA analysis for response NA.</div>

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	PC%= (SS/∑ SS) × 100
Model	0.93	13	0.072	100.28	< 0.0001	
A-Volume of eluent solvent	0.03	1	0.03	46.70	< 0.0001	14.29
B-Extraction cycles	0.02	1	0.02	26.14	0.0001	9.52
C-Elution Cycles	0.03	1	0.030	42.60	< 0.0001	14.29
D-Amount of Na$_2$SO$_4$	0.0032	1	0.0032	4.45	0.05	1.52
AB	0.00038	1	0.00038	0.53	0.48	0.18
AC	7.03E-05	1	7.03E-05	0.10	0.76	0.03
BC	0.00045	1	0.00045	0.63	0.44	0.21
BD	6.74E-05	1	6.74E-05	0.09	0.76	0.03
CD	0.00010	1	0.00010	0.14	0.71	0.05
A^2	0.092	1	0.09	129.00	< 0.0001	43.81
B^2	0.017	1	0.02	23.93	0.0002	8.09
C^2	0.019	1	0.02	25.96	0.0001	9.05
D^2	0.0015	1	0.0015	2.09	0.17	0.71
Residual	0.011	16	0.00072			
Lack of Fit	0.0097	11	0.00088	2.52	0.16	
Pure Error	0.0017	5	0.00035			
Cor Total	0.94	29				

(PC %): percent contribution, SS: sum of squares

Table 4. Comparison of the suggested technique with other methods for determination of nalidixic acid.

Method	Detection technique	LOD ($\mu g\ L^{-1}$)	Linear range ($\mu g\ L^{-}$	Ref.
Adding KMnO$_4$ in	chemiluminescence	3	100-2000	[1]
Sensor	phosphorimetry	20	60-1500	[4]
Not mentioned	HPLC	15	1000-50000	[20]
GO/ZnO PT-μSPE	spectrophotometry	0.3	1-200	This research

[a] HPLC-FD: High-performance liquid chromatography with fluorescence detection.

Table 5. Recovery and reproducibility results for determination of nalidixic acid obtained from seawater and human plasma.

Sample	Analyte added ($\mu g\ L^{-1}$)	Analyte found ($\mu g\ L^{-1}$)	Recovery	RSD (%)[a](n= 3)
Station 1, Konarak	-	1.4	-	2.9
	20	19.8	92.0	1.4
	50	49.4	96.0	3.9
Station 2, Tis	-	1.5	-	2.5
	20	19.2	88.5	4.3
	50	49.1	95.2	1.6
human plasma	-	3.2	-	3.0
	20	22.8	98.0	4.4
	50	53.1	99.8	1.1

Considering the final elution volume of 250 μL, and a sample volume of 10 mL, an enrichment factor (EF) of 40.00 for NA should be achieved. The real EF experimentally obtained was 38.80. In Table 4 the suggested method is compared to the other techniques reported in the literature for NA determination.

3.4. Determination of nalidixic acid in real samples
3.4.1. Determination of nalidixic acid in human plasma
To assess to performance of the GO/ZnO PT-μSPE technique, extraction and determination of NA in human plasma sample was carried out under optimized conditions. 250 μL of blood plasma was added to 100 μL of 6 M NaOH and then heated at 60 ^{0}C in water bath for 30 min in the first step. After cooling to 2 0C, 500 μL of 20%w/v trichloroacetic acid was added. Then, the mixture was vortexed for 1 min and afterward the sample was left for 10 min at ambient temperature for completion of reaction. Next, the solution was centrifuged at 1000 rpm at 4 ^{0}C for 10 min [19]. The archived deproteinized supernatant solution was poured into a vial and diluted to 10 mL using ultrapure water and detected in optimal conditions. NA concentration were determined as 3.20 μg L^{-1}, respectively, by a relative standard deviation (RSD, n=3) of 3.0. Moreover, plasma samples were spiked in two concentration levels to investigate the matrix effect on the accuracy of analysis. In Table 5, the results are indicated.

3.4.1. Determination of nalidixic acid in seawater
The suggested method was also employed for the determination of nalidixic acid in three seawater samples of Chabahar Bay in southern east of Iran. No salt was added for these real samples, because seawater is salt saturated by itself. In order to study the effect of sample matrix on accuracies; they were separately spiked with NA at 20 and 100 μg L^{-1} concentration levels and results are presented in Table 5. As can be seen, no serious interferences from the matrices were observed.

4. CONCLUSION
A GO/ZnO nanocomposite was synthesized and applied as an efficient sorbent for PT-μSPE of nalidixic acid in seawater and human plasma samples. Just by using a conventional spectrophotometer it made possible to precisely determine trace amounts of target compound in complicated matrices. High clean-up, excellent extraction efficiency and low organic solvent consumption are other advantages of this method. Also, the capacity of the pipette-tip by requiring only a few mg adsorbent reduced significantly the solvent consumption in the elution step, and decreased the operation time and cost.

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تعیین اسپکتروفتومتری نالیدیکسیک اسید در برخی نمونه‌های حقیقی بعد از پیش‌تغلیظ با استخراج فاز جامد در سرسمپلر به کمک جاذب نانوکامپوزیت گرافن اکسید/ روی اکسید

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چکیده

در این کار تحقیقاتی یک نانو کامپوزیت گرافن اکسید/ روی اکسید (GO/ZnO) سنتز گردید و برای استخراج فاز جامد در سر سمپلر نالیدیکسیک اسید از نمونه‌های آب دریا و پلاسمای خون انسان بکار رفت و تعیین آن با اسپکتروفتومتری بکار رفت. پارامترهای مؤثر بر روش پیشنهادی، از جمله نوع و حجم حلال شوینده، مقدار جاذب، حجم نمونه، تعداد سیکل‌های استخراج و شویش، pH محلول نمونه، نوع و مقدار نمک بررسی و بهینه‌سازی شدند. بهینه‌سازی با استفاد از روش‌های یک متغیر در یک زمان و سطح پاسخ انجام گردید. ارقام شایستگی روش به صورت زیر بدست آمد: حدّ تشخیص روش ۰/۳۰ میکروگرم بر لیتر، فاکتور پیش‌تغلیظ ۴۰ برای NA، محدودهٔ خطّی منحنی درجه بندی ۲۰۰/۰– ۱/۰ میکروگرم بر لیتر و با تکرارپذیری (RSD) بهتر از ۴/۴٪ حاصل گردید. نتایج کاربرد روش در آب دریا و پلاسما خون انسان نشان داد که این روش تجزیه‌ای می‌تواند برای تعیین آنالیت در نمونه‌های واقعی پیچیده به طور موفقیت آمیزی بکار رود.

واژه‌های کلیدی

نالیدیکسیک اسید؛ آب دریا؛ گرافن اکسید/ روی اکسید؛ استخراج فاز جامد در سرسمپلر؛ روش‌شناسی سطح پاسخ.