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# Determination of optimal adsorption-desorption conditions for selective removal of Ni(II) from petrochemical samples using ion imprinted nanosorbent

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## RESEARCH ARTICLE



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## ABSTRACT

Nanoporous particles Ni(II) ion imprinted polymer (IIP), and non-imprinted polymer (NIP) in the absence of Ni(II) ion, with 18-70 nm dimensions were synthesized, and characterized by Fourier transform infrared, energy dispersive X-ray and nuclear magnetic resonance spectroscopic methods. Then, the surface area, pore size and structural composition of the products were characterized by Brunauer-Emmett-Teller and scanning electron microscope methods. Then, modified electrodes by the IIP for Ni(II) sensing and determination, were constructed and their catalytic activity were investigated by cyclic voltammetric method. Some parameters like desorption solvent, amount of sorbent, pH and contact time were optimized, and the measurements were all conducted under optimal conditions. The optimum pH for maximum sorption was obtained 7.8. In the optimum conditions, the maximum sorbent capacity of the IIP was obtained 371.9  $\mu\text{M/g}$ . The limit of detection and relative standard deviation ( $n = 5$ ) were obtained 1.3 ng/mL and 1.47%, respectively. The pre-concentration procedure revealed a linear curve within the concentration range of 10-6000 ng/mL and a good linearity with squared correlation coefficient of  $r^2$  0.9991 was achieved. The method was applied successfully for determination of Ni(II) ion in petrochemical samples.

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## 1. Introduction

Metallic nickel is used as important material for catalysts construction in the chemical industries. Thus, it presents in industrial waste, petrochemical products and some other effluents. Therefore, determination of Ni(II) ion is considered highly important owing to its toxic characteristics and release of catalytic activity of the catalysts. However, direct determination of trace amounts of nickel(II) ion are challenging in these samples because of the complexity of the matrix and extremely low concentrations of analyte, which are often below the detection limits of available techniques [1-3].

Dimethylglyoxime (DMG) is widely used as a selective reagent for analysis of Ni(II) ion, especially for trace amounts determination of this ion by spectrophotometric methods after pre-concentration [4]. For extraction and pre-concentration some methods such as liquid-liquid extraction [5] and solid phase extraction [6] but all of these separation methods suffer from lack of selectivity. Metal ions such as Pb(II), Mn(II), Bi(III), Co(II), Cd(II), Zn(II), and Fe(II) could interfere at concentrations higher than 5 mg/L in solid phase extraction of

Ni(II) ion using DMG as a complexing agent [7]. This paper deals with a selective and simple method for separation and pre-concentration of Ni(II) ion with DMG-nanopolymer. Many sample pretreatment methods including solvent extraction, cloud point extraction, solid-phase extraction, membrane filtration, electrodeposition, flotation, co-precipitation and ion exchange have been utilized for pre-concentration of trace metals from natural waters [8-14]. Solid phase extraction is a separation technique that allows automation, miniaturization, dispenses the use of organic solvents and natural recovery of the sorbent [15].

One of the recently developed techniques for the preparation of solid-phase sorbents is the ion imprinting technique which can be used for selective separation and pre-concentration of trace metals [16]. Ion imprinted polymers (IIP) were first introduced by Nishide and his colleagues [17]. In this technique, a complex is formed through the assembly of ionic species and especial functional monomer and then is polymerized with the monomer to give the corresponding sorbent.

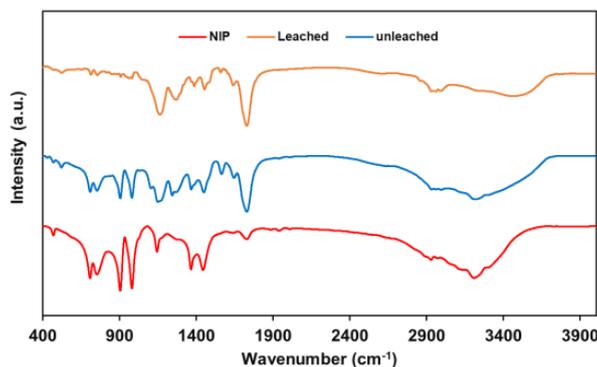


Figure 1. FT-IR spectra of the NIP, unleached and leached IIP polymers.

Cavities as templates, proper for the size and shape of the analyte, are formed in the polymer matrix upon the removal of the imprinted ion [18-21]. The IIP have been used for chromatography, sensors (such as voltammetric or potentiometric) and solid phase extraction [22-25]. The aim of the present study was to study the affinity-based separation and pre-concentration of Ni(II) in petrochemical samples in the presence of competing cations, using synthesized Ni(II) ion imprinted nanopolymer and determination with atomic absorption spectroscopy.

## 2. Experimental

### 2.1. Reagents

All HPLC grade solvents and reagents were of analytical grade, were purchased from Merck and utilized without any further purification.

### 2.2. Apparatus

A Varian AA240FS Fast Sequential Atomic Absorption Spectrometer (AAS) was used for the determination of metal ions. A PerkinElmer Lambda 35 model, double beam UV-Vis spectrophotometer was utilized to measure the spectra of the complex. The prepared IIP were studied using a FT-IR instrument (M-500 Fast-Scan IR Spectrometer Buck Scientific, East Norwalk, CT 06855, United States) and scanning electron microscopy (S-4160 Hitachi). A Bruker Avance 300 spectrometer was used for NMR studies. The elemental composition was determined by energy-dispersive X-ray spectroscopy, Samx. Surface area analysis was achieved using a micromeritics TriStar II PLUS instrument. A Metrohm 713 pH meter was used for pH adjustments. Stirring of the solutions was accomplished by a Heidolph MR3001 magnetic stirrer (Schwabach, Germany).

### 2.3. Preparation of Ni(II) ion imprinted polymer

In this work; complexation was carried out by adding DMG (2 mmol) and nickel (1 mmol), as the ligand and imprinted metal ion, respectively, to ethanol (10 mL) as the porogen solvent in a 50 mL vial with stirring for 15 min. Then, the resulting Ni(II)-DMG complex solution was mixed with EGDMA (16 mmol), MAA (4 mmol) and benzoyl peroxide in toluene (0.1 mmol), as the crosslinker, functional monomer and free radical initiator, respectively. The mixture was stirred at room temperature. The polymerization mixture was cooled to 10 °C and purged with nitrogen gas for 15 min, and thermally polymerized in a bath at 60 °C for 12 h. After polymerization, the excess amount of the solvent was removed by centrifuga-

tion of the resulting suspension solution. The prepared polymer was washed several times with methanol:ethanol (1:2, v:v) to remove the unreacted materials and then was treated with 3.0 M HCl solution to leach the imprinted metal ion until the wash solution was free from Ni(II) ions. Finally, it was washed with double distilled water until a neutral pH was reached and dried at 60 °C. Non-imprinted polymer (NIP) was also prepared in a similar way to that of the IIP preparation with no using Ni(II) ions [26].

## 3. Result and discussion

### 3.1. Characterization of polymers

The FT-IR spectra of the non-imprinted, leached and unleached ion imprinted polymers are presented in Figure 1. In the IR spectra, absorption bands related to C-OH (3440 cm<sup>-1</sup>), C=N (1564 cm<sup>-1</sup>), C=N<sup>+</sup>-O<sup>-</sup> (1550-1650 cm<sup>-1</sup>), and C-H bands (1366, 1448 cm<sup>-1</sup>) are appeared. In the IR spectrum of Ni<sup>2+</sup>-DMG IIP (Figure 1), the  $\nu_{(C=N)}$  in the free DMG and NIP has changed which can be an indication of binding Ni<sup>2+</sup> through the nitrogen atoms. However, the C=N and N<sup>+</sup>-O<sup>-</sup> stretching vibrations at 1564 and 1645 cm<sup>-1</sup> in the unleached material were shifted to 1640 cm<sup>-1</sup> in leached IIP, implying that the C=N and N<sup>+</sup>-O<sup>-</sup> groups in DMG are involved in Ni<sup>2+</sup> ion bonding. No bands appeared in the region of 1550-1650 cm<sup>-1</sup>, suggesting the absence of C=N<sup>+</sup>-O<sup>-</sup> (imine oxide) groups in the NIP materials. The IR spectrum of Ni<sup>2+</sup>-DMG IIP also shows the presence of OH deformation band at 1728 cm<sup>-1</sup> which shows the formation of a hydrogen bridge. This hydrogen bridge confirms the formation of the red square planar Ni<sup>2+</sup>-DMG IIP and is not observed in the NIP.

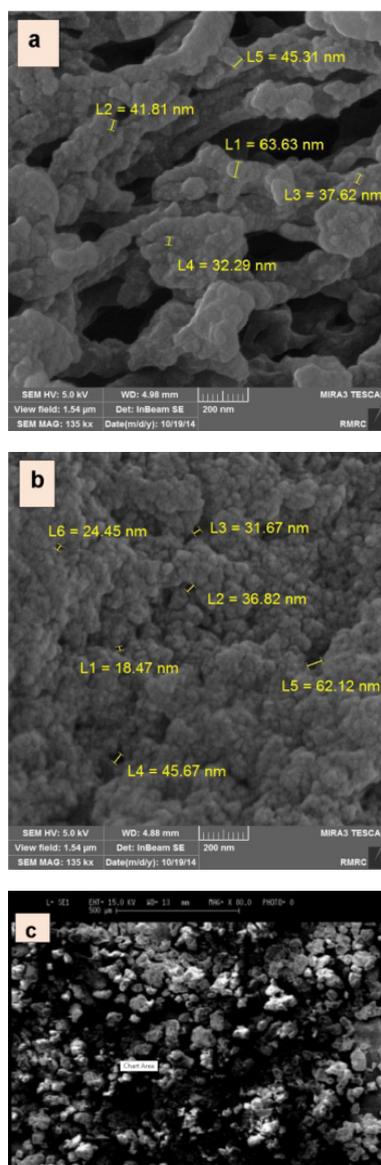
The <sup>1</sup>H- and <sup>13</sup>C-NMR spectra of the IIP polymers were agree with suggested structure (<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>,  $\delta$ , ppm): 1.910 (s, 3H, CH<sub>3</sub> methyl), 11.320 (s, 1H, OH<sub>alcohol</sub>); <sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>,  $\delta$ , ppm): 9.46 (CH<sub>3</sub> aliphatic), 153.26 (C imine)).

The surface structure of unleached, leached IIP and NIP was shown in Figure 2, respectively. As seen, the IIP exhibited high porosity which is considered an important feature in the extraction procedures since it improves the interactions between the analytes and the absorbent.

The elemental composition of the leached and unleached IIP particles was investigated by energy-dispersive X-ray spectroscopy (EDX). The SEM images show that the porosity is changed after removal of the Ni(II) ions (Figure 2). The EDX results in Figure 3 show that 21.3 wt% of Ni(II) is present in the unleached polymer particles, whereas the spectra of the leached particles show a negligible Ni(II) peak. Furthermore, the percentage weights of carbon, oxygen and nitrogen is quite the same, confirming that during leaching (using 3.0 M HCl) only the Ni<sup>2+</sup> ions are leached out and not the ligand [27].

**Table 1.** Surface area analysis data of NIP, unleached and leached IIP nanoparticles.

Parameter	Unleached IIP	Leached IIP	NIP
Multipoint BET surface area (m <sup>2</sup> /g)	18.1	26.3	0.8

**Figure 2.** SEM images of the unleached (a), leached IIP polymers (b) and NIP (c).

### 3.2. Electrochemical behavior of the modified carbon paste electrode (MCPE) with IIP

Cyclic voltammetry (CV) were performed to elucidate the catalytic activity of IIP-MCPE toward Ni(II) ion (Figure 4). The CV was run starting from -0.5 to 1.0 V and back (Scan rate: 100 mV/s). As can be seen, the CV signal of the IIP-MCPE is higher than that of the NIP. This indicates that the presented IIP in the electrochemical sensor intensively absorbs Ni(II) ions from the aqueous solution [28].

According to Table 1, the Brunauer, Emmett and Teller (BET) surface areas of the leached particles are higher than those of the unleached particles.

### 3.3. Effect of pH

The effect of pH on the determination of the Ni(II) ion is shown in Figure 5. Among the investigated parameters, pH is the most important factor for adsorption of Ni(II) ion on the IIP particles [27]. Therefore high concentration of the hydrogen ions interfere with the rebinding of the metal ion to the cavity as the hydrogen ions also bind with the cavity, whereas a low concentration of hydrogen ions and existence of OH<sup>-</sup> usually leads to the formation of precipitates with ligands. To estimate the effect of pH on the sorption efficiency, the pH of 10 mL of sample solutions containing 1 μg/mL of Ni(II) ions was adjusted in the range of 1-10 (Figure 5). Therefore, for further studies, all the samples were buffered to pH = 7.8±0.1 by adding phosphate buffer [29].

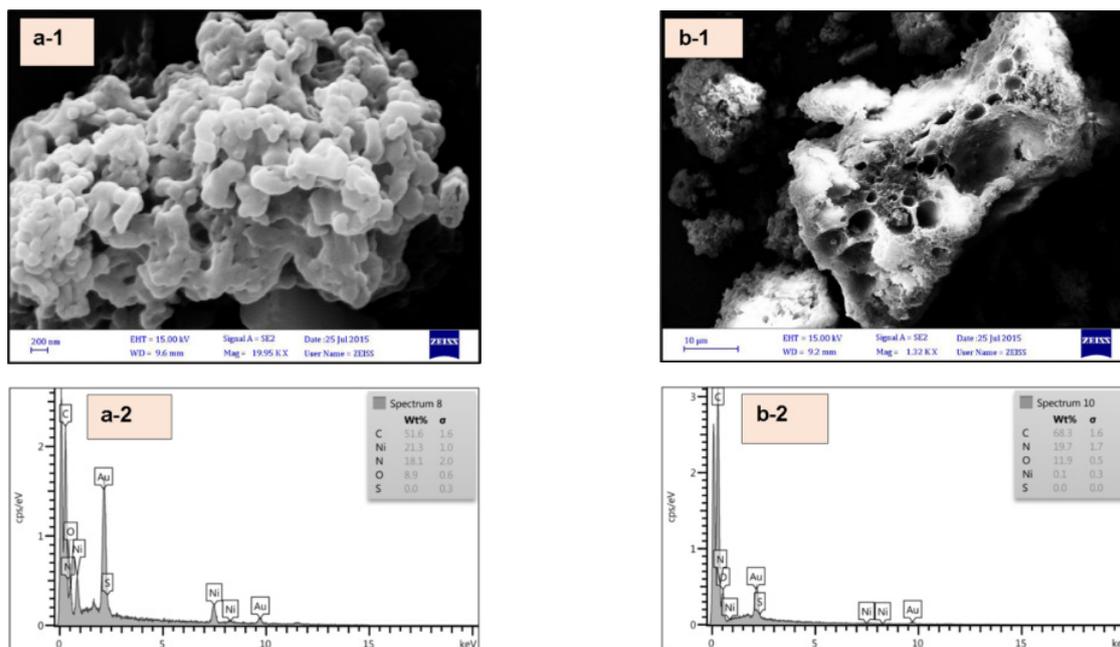


Figure 3. EDX images and spectra of the unleached (a-1, a-2) and leached IIP particles (b-1, b-2).

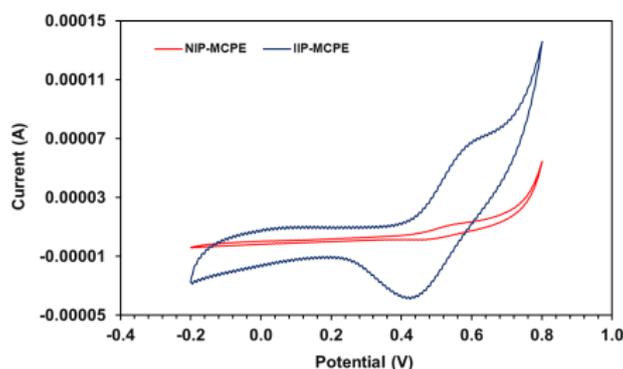


Figure 4. Cyclic voltammograms of Ni(II) ions (Metals concentration:  $4 \times 10^{-6}$  M; pH = 7.8).

### 3.4. Effect of time

The time effect on the sorption of Ni(II)-IIP and NIP particles were studied at varying time values. As seen from Figure 6, an optimum sorption time of 20 min was obtained for the quantitative sorption of Ni(II) ions from the solution into the IIP particles.

### 3.5. Effect of sorbent weight

To investigate the optimum sorbent weight, 5-80 mg of leached IIP particles was added to 10 mL of  $1 \mu\text{g/mL}$  Ni(II) ion solution at pH = 7.8. The obtained suspensions were magnetically stirred for 20 min and then the suspensions were centrifuged. After that the content of the solutions was determined by AAS. As seen, 40.0 mg of leached IIP was found to be the optimal amount of the IIP to achieve the maximum recovery.

### 3.6. Effect of eluent

A series of eluents, including  $\text{HNO}_3$ , HCl, Aqua regia,  $\text{H}_2\text{SO}_4$  and  $\text{C}_3\text{H}_6\text{O}_2$  (Acetic acid) were used for elution of Ni(II) ions from imprinted polymers. It was found that HCl (3.0 M)

provided the most effective elution of Ni(II) ions from sorbent. The effect of eluent volume on the recovery of nickel(II) ions was also studied. Therefore quantitative desorption was obtained with 10 mL of HCl [27].

### 3.7. Selectivity studies

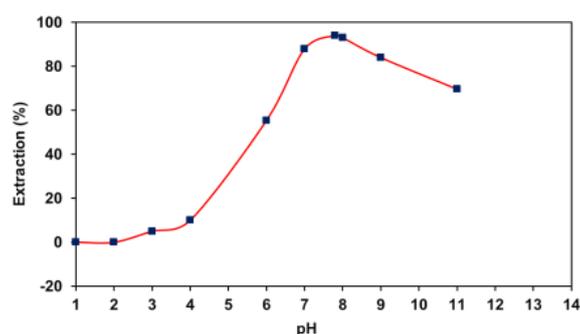
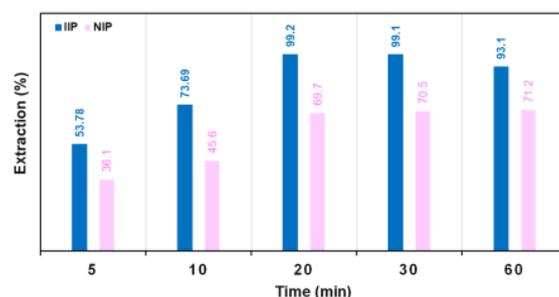
The selectivity of the sorbent over the other selected alkali metals, alkaline earth metals and transition metals, coexisting with the nickel ions, were studied. The selection of interfering metal ions for this study was based on the similarity of chemical properties of other metals to the nickel(II) ions [27]. The selectivity of the Ni(II)-IIP for nickel in this study was much better compared to that was previously reported for some polymers [30,31]. Zn(II), Fe(II), Co(II), Cu(II), Ca(II), Mg(II), Na(I) and K(I) in the petrochemical samples did not interfere with the binding of Ni(II) on the IIP as shown in Table 2. Despite the fact that Fe(II) and Co(II) [32] are known to interfere with the complexation of nickel with DMG, these metals did not show any interference with the extraction efficiency of Ni(II) when the IIP nanoparticles was used. This strongly suggests that the IIP nanoparticles can be effectively used as an absorber for nickel(II) ions (Table 2).

**Table 2.** Extraction (E), distribution ratio (D), selectivity coefficient ( $\alpha$ ) and relative selectivity coefficient (K) values of IIP and NIP for selected metal ions and nickel(II) ions.

Metal ion	E <sub>IIP</sub> (%)	D <sub>IIP</sub> (mL/g)	E <sub>NIP</sub> (%)	D <sub>NIP</sub> (mL/g)	$\alpha_{IIP}$	$\alpha_{NIP}$	K
Ni(II)	78	886.3	43.4	192.2	-	-	-
Fe(II)	40	166.6	13.3	38.6	5.3	4.9	1.0
Co(II)	50	250.0	31.8	117.1	3.5	1.6	2.1
Cu(II)	35	134.6	20.2	63.5	6.5	3.0	2.1
Zn(II)	25	83.3	22.0	70.7	10.6	2.7	3.9
Ca(II)	15	31.8	10.1	28.1	27.8	6.8	4.0
Mg(II)	20	62.5	17.2	52.1	14.1	3.6	3.8
Na(II)	13	28.3	8.0	21.7	31.3	8.8	3.5
K(II)	9	24.7	7.6	20.6	35.8	9.3	3.8

**Table 3.** Determination of nickel(II) ions in petrochemical samples (n = 4).

Sample	Ni(II) ion content (ng/mL)		Recovery (%)
	Added	Found	
Petrochemical sample 1	0	8.6±1.4	-
	10	18.5±1.6	99.4
Petrochemical sample 2	0	12.7±2.1	-
	10	22.4±1.9	98.6

**Figure 5.** Effect of pH on the sorption of Ni(II) ions using IIP polymers.**Figure 6.** The time effect on the sorption of Ni(II)-IIP and NIP particles.

### 3.8. Method performance

The precision of the method for five standard solutions containing 1  $\mu\text{g/mL}$  of Ni(II) ions was assessed by determining the relative standard deviation and was found to be 1.47% [27]. The detection limit defined as  $C_{LOD} = 3 S_b/m$ , where  $S_b$  is the standard deviation of four replicate blank signals for a sample volume of 10 mL and  $m$  is the slope of the linear section of the calibration curve after pre-concentration, were found to be 1.3 ng/mL. The pre-concentration procedure revealed a linear curve within the concentration range 10 to 6000 ng/mL. A good linearity with squared correlation coefficient ( $r^2$ ) 0.9991 was achieved.

### 3.9. Real sample analysis

The feasibility of the Ni(II)-IIP sorbent for the determination of Ni(II) ions in real samples was verified by the analysis of standard Merck samples. The results of analysis show that, the metals ions recovery is quantitative. Standard addition method was employed for determining the concent-

rations of target metal ions. The results for the determination of Ni(II) ions in petrochemical samples are summarized in Table 3.

### 3.10. Comparison with other pre-concentrating sorbents

The adsorption capacity of this work is compared with those of other pre-concentrating sorbents in Table 4.

## 4. Conclusion

In the present study, new Ni(II) imprinted polymer nanoparticles were prepared and used for the determination of nickel(II) ions from petrochemical samples. The obtained results indicated that by combining affinity-based IIP extraction and a simple AAS method without applying a derivatization step, a precise, selective and sensitive determination of nickel(II) ion in low concentration would be possible. In addition, various parameters of the structure of the polymer particles and the optimum conditions for the pre-concentration were investigated.

**Table 4.** Comparative data from previously reported solid-phase extraction method for nickel(II) ions separation.

Adsorbent	Adsorption capacity ( $\mu\text{mol/g}$ )	References
Nickel(II)-5-vinyl-8-hydroxyquinoline ion-imprinted polymer	33.7	[32]
Nickel(II)-imprinted amino-functionalized silica gel sorbent	214.7	[33]
Nickel(II)-dithizone ion-imprinted polymer	21.9	[34]
Natural kaolinite	28.42	[35]
Nickel(II)-8-hydroxyquinoline ion-imprinted polymer	22.9	[36]
Nickel(II)-methacryloylhistidinedihydrate ion-imprinted polymer	177.9	[37]
Nickel(II)-N-vinyl-2-pyrrolidone ion-imprinted polymer	57.8	[38]
Nickel(II)-DMG ion-imprinted nanopolymer	371.9	This work

The maximum sorbent capacity of the proposed Ni(II)-IIP sorbent was compared with previously published maximum sorbent capacities for other sorbents and was shown to be higher than the published values.

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Conflict of interests: The authors declare that they have no conflict of interest.

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