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#### Iron Ionic Imprinted Polymers IIps for Separation and Preconcentration of Iron from Crude and Fuel Oil

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#### <u>Abstract</u>

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A novel Iron ion-imprinted polymers (IIPs) was synthesized by bulk polymerization using different types of monomers such as 1-vinyl imidazole and Styrene, respectively. Molar ratios of monomer, template and cross-linking agent for polymerization, various monomers and solvents were studied to obtain the largest adsorption capacity for Iron. The prepared Iron-IIPs were characterized using energy dispersive X-ray spectroscopy (EDX), Fourier - transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM).

The three-dimensional network structure surfaces of Iron-IIPs are unaffected by the elution procedure. Iron ions were successfully eluted from IIPs using a mixed solution from ethanol and acetic acid. The maximum adsorption capacity of Iron-IIPs was is  $(514.5)\mu$ mol/g for Iron-IIP1(using styrene as a monomer) and (429.1)  $\mu$ mol/g for Iron-IIP2(using 1-vinyl imidazole as a monomer). The adsorption by Iron-IIPs followed a Langmuir isotherm models. Solid-phase extraction (SPE) syringe packed with ionic imprinted polymers (IIPs) were used to selective separation for Iron ion from Crude or fuel oil and digest the polymer to determination the Iron by flame atomic absorption spectroscopy (FAAS) Abbreviation IIP-SPE-FAAS.

Keywords: Ionic imprinted polymer, SPE-FAAS, adsorption, crude oil, fuel oil.

#### 1. Introduction

Trace element detection is critical in the petroleum industry since it informs geologists about crude oil sources, movement, and kinds [1]. Metallic elements are naturally present in crude oil, and they come from the plants that were transformed to oil, as well as the rocks and soils that were present near the oil throughout its formation. Contamination of crude oil during storage or transportation owing to contact with, and corrosion of tanks or pipes is another



source of metals in crude oil [2, 3]. The presence of metallic elements in oil and gasoline over specific amounts is undesirable because they can harm engine components by producing deposits and corrosion, or they can cause fuel deterioration. The emission of pollutants into the atmosphere as a result of burning fuel is linked to health hazards [4, 5]. Iron can cause corrosion in boilers and furnaces during oil processing and be transported to distillation products, such as diesel fuels and gasoline. It's a poisonous catalyst used in oil refining [6]. The routine continuous monitoring and determination of Iron in crude oil and its distillation products is therefore essential [7, 8]. However, determination of Iron concentration in water still a difficult problem due to detection limits of instruments, low concentration, and interference from matrix sample [9].

Several methods are used are used for the determination of trace metals in crude oil, such as inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma optical emission spectrometry (ICP OES), graphite furnace atomic absorption spectrometry (GFAAS) and flame atomic absorption spectrometry (FAAS) [10,11]. However, ICP-based approaches, in particular, cannot tolerate a large organic load and must be converted into a format that can be delivered into the instrument without producing issues. Preparing such complicated and thick samples as crude oil is difficult, and the most typical method is digestion, which takes time, is prone to contamination and analyst loss, and necessitates the use of toxic and corrosive acids [12.13]. To boost the Iron concentration, several separation and pre-concentration procedures were examined. Liquid-liquid extraction (LLE), solid phase extraction (SPE), and chemical precipitation techniques can all be used to separate metal ions. SPE is one of them, with various advantages like as cheap cost, simplicity, and time savings for analysis [14].

Ion imprinted polymers (IIP) have recently drawn the interest of numerous researchers due to their strong selectivity for imprinted ions and their ability to be used in natural complex matrixes. Continuous advances in analytical chemistry have recently resulted in the creation of novel chemical separation methods that are critical. The development of novel extractants and adsorbents has been fueled by the demand for a more selective system for metal ion separation [15, 16]. SPE is a simpler and faster method. Compared to classic liquid–liquid extraction, this method is more cost-effective and environmentally friendly. The limited selectivity of the retention mechanism is the main issue with SPE columns filled with conventional stationary



phases. Using columns filled with materials based on molecularly imprinted polymers (MIPs) as selective sorbents, a desired level of selectivity may be achieved [17-20]. Specific identification enables molecular imprinting as a sort of unique technology. The template molecules are mixed with functional monomers first to create a molecularly imprinted polymer. The crosslinking agents and initiators are then added to begin the polymerization process. The template molecules are then removed via a chemical or physical procedure. Certainly, a molecularly imprinted polymer with a large number of space voids that correspond to the destination molecules is fully created [21-23]. The manufactured sorbent for metal ions is known as ion-imprinted polymer (IIP), which is similar to MIPs but detects inorganic ions after imprinting [24-25]. Various methods, such as bulk polymerization [26], suspension polymerization [27] precipitation polymerization [28], sol-gel polymerization [29,30] and surface imprinted technique [31, 32] have been used for the preparation of ion imprinted polymers.

In this work, Iron-IIP was synthesized by bulk polymerization using Iron(III) chloride hexahydrate (ferric chloride hexahydrate) as a templet with styrene and 1-vinyl imidazole as monomer to prepare two types of Iron-IIPs with ethylene glycol dimethacrylate (EGDMA) as crosslinker and benzyl peroxide as initiator for removal of Iron ions from crude and fuel oil. Effects of monomer and EGDMA dosages on adsorption performance were studied. Adsorption behaviours with various functional monomers, cross-linking agents and solvents were also investigated. SEM, FTIR, and EDX were used to characterize the prepared IIPs. Furthermore, effects of adsorption conditions, such as flow rate of solution pass-through solid phase extraction syringe, adsorption time and initial Iron concentration on adsorption capacity were investigated.

#### 2. Experimental

#### 2.1 Reagents and Chemicals

Styrene, 1-vinyl imidazole, ethylene glycol dimethacrylate (EGDMA) and benzyl peroxide and were purchased from Sigma–Aldrich (St. Louis, MO, USA, <u>www.sigma-aldrich.com</u>), chloroform, methanol, acetic acid, acetonitrile, and Iron (III) chloride hexahydrate (ferric chloride hexahydrate) FeCl<sub>3.</sub>6H<sub>2</sub>O were purchased from Merck (Darmstadt, Germany,www.merck.com). Nitrogen gas (99.99%) from Arab gulf factory Baghdad.

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#### 2.2 Instrument

The control was performed using atomic absorption spectrophotometer pginstrument (England), the use of UV 1800pc spectrophotometer (Shimadzu, japan), scanning electron microscopy SEM ZEISS (United States), EDX MIRA3 TESCAN (Czechoslovakia), FTIR FTIR 8000 (Shimadzu, Japan) and ultrasonic (W.GERMANY) was used to stir up the copolymer solution.

#### 2.3 Preparation ionic imprinted polymer

For preparation first Iron molecularly imprinted polymer (Iron-IIP1), 1mmol (0.27 g) from Iron (III) chloride hexahydrate FeCl<sub>3.6</sub>H<sub>2</sub>O dissolved in little amount of methanol then mixed with 20 mmol (2.08 g) styrene as the monomer, after that added 40 mmol (7.92 g) ethylene glycol dimethacrylate (EGDMA) to the solution as the crosslinker, followed that 0.3 mmol (0.07 g) benzoyl peroxide dissolved in 3ml chloroform and add to the mixture as the initiator. All these materials were dissolved in 5  $\pm$  1ml methanol (CH3OH). While the second Iron-MIP2 were achieved by mixed the same amount of Iron (III) chloride hexahydrate FeCl<sub>3.6</sub>H<sub>2</sub>O as the template with 20 mmol (1.88g) 1-vniyl imidazole as the monomer after that added 40 mmol (7.92 g) ethylene glycol dimethacrylate (EGDMA) to the solution as the crosslinker and 0.3 mmol (0.0 g) benzoyl peroxide as the initiator which dissolved in 5  $\pm$  1ml of methanol (CH3OH). For obtaining a homogeneous solution, the mixture was shake for 5 minutes. N2 passes for 30 minutes on the mixture to extract oxygen from the solution. then, the solution was placed in a water bath at 60° C overnight. When the reaction was finished, the molecularly imprinted polymer became solid, and the polymer was dried and smashed to yield a polymer particle. The templet extract from MIP by soxhlate in CH3OH/CH3COOH (10:1 v/v).

Respectfully, Non-molecularly imprinted polymers are made using the same ingredients and circumstances as Iron-IIP1 and Iron-IIP2, but without the Iron (III) chloride hexahydrate FeCl<sub>3</sub>.6H<sub>2</sub>O (template). The same distribution was used in the preparation of non-imprinted polymers (NIPs), but without the template.

#### 2.4 Sampling Procedure

Prepare a stock solution 1000 ppm of Iron in organic medium by dissolved (1.205 gm) from Iron (III) chloride hexahydrate  $FeCl_3.6H_2O$  in little amount of methanol and add base oil to complete volume 250 ml and use to prepare a series of concentration (1, 50, 100, 200, 300, 400,

500, 600, 700, 800 ppm). 1 ppm solution diluted to prepare (0.1, 0.5, 1, 2, 3, 4, 5, 10, 15, 20, 25 ng/ml) for preconcentration method to determination the little amount note detect direct in flame atomic absorption spectrophotometer. Also, brought several samples of crude and fuel oil.

#### 2.5 The Sampling Device

Each syringe was loaded with varied weights (0.1 and 0.2gm) of IIPs in a 10 ml solid phase extraction syringe.

#### 2.6 Extraction and digestion procedure

Iron ion was extracted from synthetic solution, diluted synthetic solution and crude or fuel oil using Iron-IIP1 (styrene as a monomer) and Iron-IIP2 (1-vinyl imidazole as a monomer) by solid phase extraction (SPE) syringe. This syringe was prepared by packing it with an IIP 0.2 mg, the size of its container 10 ml. The solution containing Iron pass through SPE syringe by vacuum process using peristaltic pump in different rate.

IIP was collected from column in the small beaker, dried for 60 minutes, than a 1mL of concentrated sulfuric acid is added to it and left for a 8 minute, the next step concentrated nitric acid 1ml is added to it and heated at a 60 temperature after that added deionized water to the mixture, later estimated directly by flame atomic absorption spectrophotometer.

#### 3. Result and Dissection

#### 3.1 Characterizations

#### 3.1.1 FT-IR

Spectral analysis was used to determine the interaction between Iron ion and monomer. The Cesium Iodide (CsI) pellet technique registered the FT-IR spectrum in the range of 250–4000 cm<sup>-1</sup>. Figure (1) shows the FT-IR spectra of Iron-IIP1 before and after elution (1). Both spectra exhibit comparable backbones, indicating that the elution procedure has no effect on the fundamental polymer network structure. The spectrum of Iron-IIP1 before elution has strong bands at 1554.52, 815.83, corresponding to Fe-O and Fe-Cl groups, which indicates that Iron (III) chloride hexahydrate had participated in the synthesis of Iron-IIP1. However, these tow peaks disappear after eluting Fe ions due to destruction of original interaction between styrene and Fe, indicating that Fe had been removed successfully.

The FT-IR spectra of Iron-IIP2 are shown in Figure (2) where a appears band at 1552.95,



813.37cm<sup>-1</sup> corresponding to Fe-O and Fe-Cl groups. These two peaks disappear after eluting Iron ions due to the destruction of original interaction between 1-vniyl imidazole and Iron. The main peaks obtained from figures list in the Tables (1) and (2).

#### 3.1.2. SEM

Morphological analysis is an important characteristic for understanding the size and arrangement of areas where Iron ion was removed. SEM images were used to assess the morphology of the Fe-IIPs. For Iron-IIP1 Figure (3) (A, B) shows the surface morphologies of the particles before and after elution. It can be seen that the surface of eluted Iron -IIP1 is containing pores and rougher than that of non-eluted one as a result of the removing of Fe ions. Surface morphologies of Iron -IIP2 before and after elution shown in Figure (4) (A, B). For two types of preparation Because of the well-defined binding sites on the imprinted polymer, IIPs has a lot of holes on the surface, making it a good sorbent. In addition, the images shows that the diameters of pores ranging between (300-350 nm) for Iron -IIP1 and (200-250 nm) for Iron - IIP2 are uniform in sizes and shapes (irregular).



Fig. (1): FTIR spectrum of Iron -IIP1 (Using styrene as a monomer). A) Before the removal of Fe ion B) After the removal of Fe ion

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Fig. (2): FTIR spectrum of Iron -IIP2 (Using 1-vinyl imidazole as a monomer). A) Before the removal of Fe ion B) After the removal of Fe ion

Table (1) The FTIR Spectrum's most well-known peaks Iron -IIP1 using (styrene) as a
functional monomer

NO	Eurotional Crown	$\mathbf{F}_{\mathbf{a}}\mathbf{C}$ ( $\mathbf{a}\mathbf{m}^{-1}$ )	Iron-IIP2 before templet	Iron-IIP2 after templet
NU	r unctional Group	$\mathbf{FeCl}_{3}\mathbf{OH}_{2}\mathbf{O}\left(\mathbf{Cm}\right)$	removal (cm <sup>-1</sup> )	removal (cm <sup>-1</sup> )
1	C II alimbatia		2904.60	2927.74
1	C-H anphatic		2962.46	2991.39
2	C=O ester		1731.96	1728.10
3	C-H aromatic		3137	3037.68
4	C=C aliphatic		1600	1558.38
5	C=C vinyl		1649.02	1643.24
6	C-0		1263.29	1267.14
7	C-N		1475.44	1475.44
8	Fe-O	1554.52	1552.59	
9	Fe-Cl	815.83	815.13	



### Table (2) The FTIR spectrum's most well-known peaks Iron -IIP2 using (1-vinyl)

imidazole) as monomer

NO Functional Group		$\mathbf{FoCl} \mathbf{6HO}(\mathbf{am}^{-1})$	Iron-IIP1 before	Iron-IIP1 after template
NO	r uncuonar Group	reci <sub>3.0112</sub> 0 (cm )	template removal (cm <sup>-1</sup> )	removal (cm <sup>-1</sup> )
1	C-H aliphatic		2983.67	2952.81
	C-11 anphate		2958.60	2987.53
2	C=O ester		1724.24	1730.03
3	C-H aromatic		3026.10	3028.03
4	C=C aliphatic		1608.52	1606.59
5	CH <sub>2</sub> =CH <sub>2</sub> aromatic		1635.52	1633.59
6	C-O		1296.08	1296.08
7	Fe-O	1554.52	1554.52	
8	Fe-Cl	815.83	815.83	



Fig. (3): SEM of Iron-IIP1 (Using styrene as a monomer). A) Before the removal of Iron ion, B) After the removal Iron ion

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Fig. (4): SEM of Iron-IIP2 (Using 1-vinyl imidazole as a monomer). A) Before the removal of Iron ion, B) After the removal Iron ion

#### 3.1.3. EDX

The EDX test was performed to assess the composition of Iron-IIPs and to confirm that the Iron ion was effectively eluted. Figures (5 and 6) show the EDX spectra of Iron -IIPs before and after elution. The results in Figure (5A and 6A) confirm the presence Iron ion in the structure. As shown in Figure (5B and 6B), Iron ion is not present in the structure of Iron-IIPs after elution, indicating that the elution of Iron ion with porogen solvent CH3OH/CH3COOH (**10:1**  $\mathbf{v/v}$ ) successfully. Several experiments were conducted to determine o the optimal ratios of Iron ion: monomer: cross linker for preparing molecularly imprinted polymers and non-imprinted polymers. Table (3) shows the ideal ratios for creating MIPs and NIPs with the best performance characteristics.



Fig. (5): EDX spectrum of Iron-IIP1 (Using styrene as a monomer). A) Before the removal of Iron ion B) After the removal of Iron ion

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Fig. (6): EDX spectrum of Iron-IIP2 (Using 1-vinyl imidazole as a monomer). A) Before the removal of Iron ion. B) After the removal of Iron ion.

#### **3.2 Adsorption Condition**

#### 3.2.1. Adsorption time

In the industrial use of produced IIPs, the adsorption rate is a critical factor. The effects of adsorption capacity on adsorption time (1, 3, 5, 10, 30, 60, and 120 minutes) were investigated, with the findings for two IIPs given in Figure (7). The adsorption capacity increases dramatically in the first 5 minutes, showing that the adsorption of Iron ion in organic solution with produced Fe-IIPs is quite rapid at initially. Then, as the adsorption duration is increased from 5 to 10 minutes, the adsorption capacity improves somewhat before remaining constant after 10 minutes. Adsorption kinetics show that the binding sites in prepared Iron-IIPs have a high affinity for Fe ions, resulting in high adsorption efficiency.

Table (3) The deviation ratios of templet, monomer, crooslinker and solvent in thepreparation of Iron - IIPs and NIPs

NO. of IIP	Ratio	salt FeCl <sub>3</sub> .6H <sub>2</sub> O	Monomer Styrene	Cross linker EGDMA	Initiator	Solvent	Result
IIP1	%	15.75	40.92	43.01	0.3	6ml CH3OH	Salt Precipitate
	mmole	3.70	9.61	10.10	0.3	0	at the bottom
IIP1	%	3.23	31.09	65.36	0.3	6ml CH3OH	Pile white gel
	mmole	1	9.61	20.20	0.3	0	a no mano gor
IIP1	%	1.73	32.72	65.25	0.3	6ml CH3OH	White rigid
	mmole	1	20.0	40.0	0.3	0	······
NIP1	%		7.010	92.63	0.3	6ml CH3OH	White rigid
	mmole		0.3	3.964	0.3		·······

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		salt FeCl <sub>3</sub> .6H <sub>2</sub> O	Monomer 1-vinyl imidazole	Cross linker EGDMA	Initiator	Solvent	Result
IIP2	%	15.09	43.38	41.21	0.3	6ml CH3OH	Salt Precipitate
111 2	mmole	3.7	10.63	10.10	0.3		at the bottom
IIP2	%	3.13	33.29	63.27	0.3	6ml CH3OH	Salt Precipitate
111 2	mmole	1	10.63	20.20	0.3		at the bottom
IIP2	%	1.63	32.62	65.25	0.3	6ml CH3OH	White rigid
111 2	mmole	1	20.0	40.0	0.3		,, inte rigiu
NIP2	%		7.010	92.63	0.3	6ml CH3OH	White rigid
INIF 2	mmole		0.3	3.964	0.3		, interigiu



Fig. (7): Time-dependent changes in adsorption capacity (20 mL 50 mg.L-1 Iron solution, 100 mg IIPs, 25°C).

## A) Iron-IIP1 (Using styrene as monomer B) Iron-IIP2 (Using 1-vinyl imidazole as monomer)

#### 3.2.2. Maximum adsorption capacity

Appraise the ad sorption achievement of Iron -IIPs, the effect of initial Iron ion concentration ranging from (0.178) to (12.5)  $\mu$ mol/ml on adsorption capacity was studied using the following equation [33].

$$Q = \frac{(C_i - C_f)V_s \times 1000}{M_{MIP}}$$
 ----- (1)



Where Q is the binding capacity of MIPs ( $\mu$ mol/g), C<sub>f</sub> is the final Iron concentration ( $\mu$ mol/ml), Ci is the initial Iron concentration ( $\mu$ mol/ml), Vs is the volume of solution tested (ml) and *M*MIP is the mass of dried polymer (mg).

The adsorption capacity increases dramatically at initially, then steadily increases as the concentration of Iron ion rises, as seen in Figure (8). The mass transfer driving force operates by increasing the differential between the concentration of Fe ion in bulk solution and around the surface of IIPs as the initial Iron) ion concentration rises, resulting in a large increase in equilibrium adsorption capacity. When the iron concentration is more than 8 mol/ml, however, the quantity of adsorbed metal remains constant.

Tables (4) and (5) shows the adsorption capacity if Fe -IIP1 and Iron -IIP2 respectively. Langmuir isotherm models may be used to calculate Iron -IIPs' maximal adsorption capacity. Shown in Figure (9). The maximum adsorption capacity is (514.5)  $\mu$ mol/g for Iron -IIP1 and (429.1) $\mu$ mol/g for Iron -IIP2. The Iron -IIPs prepared in this work has the high adsorption capacity. The uniform and attainable imprinted binding sites greatly enhance the adsorption execution of Iron ions by the prepared Fe-IIPs. Moreover, the binding between monomers and Iron is very stable, allowing for simpler chelation formation. The Langmuir adsorption isotherm equation shown in the following [33].

Where Q is the binding capacity,  $Q_{max}$  is the maximum apparent binding capacity,  $C_{free}$  is the free analytical concentration at equilibrium (µmol/L), and Kd is the dissociation constant at binding site. In a linear plot of Q/C<sub>free</sub> vs. Q, the equilibrium dissociation constant was estimated from the slopes, and the apparent maximum number of binding sites was derived from the y-intercepts.

#### 4. Application

#### 4.1 Determination of Fe ion in synthetic organic solution

Series of synthetic solution was prepared to study the recovery of SPE-FAAS by using Iron-IIPs shows in Table (6).



#### 4.2 Preconcentration in organic solution

Preparations of 100 ml of sample containing 5.0 ng/ml of Iron ion by using base oil were loaded into the Iron-IIPs SPE syringe at 1.0 ml/min. After the sample loading, for 5 minutes, air was circulated through the column. Then, Iron-IIP was collected from column in the small beaker and add 1mL of concentrated sulfuric acid left for a 8minute. After that concentrated nitric acid 1ml is added to it and heated at a 60 temperature and added distilled water to the mixture to complete volume to 5ml, later estimated directly by flame atomic absorption spectrophotometer. The loading of 100 ml solution sample with a digest volume 5ml



Fig. (8): The relationship between adsorption capacity and initial Fe ionA) For Iron-IIP1B) For Iron-IIP2



Fig. (9): The relationship between Q/C<sub>free</sub> (L/g) and adsorption capacity to calculate maximum adsorption capacity A. for Iron-IIP1 B. for Iron-IIP2



# Table (4) The results of the adsorption capacity experiments for Iron-IIP1 and their relationship with the initial concentration

Ci	C <sub>f</sub>	Ci-C <sub>f</sub>	O(um al/a)	$O(C_{1}/a)$	
(µmol/ml)	(µmol/ml)	(µmol/ml)	Q (µmor/g)	$\mathbf{Q}_{\mathbf{U}} \mathbf{C}_{\mathbf{f}} (\mathbf{L}_{\mathbf{f}} \mathbf{g})$	
0.178	0	0	0	0	
0.892	0	0	0	0	
1.785	0	0	0	0	
3.571	1.339	2.232	223	0.166	
5.357	2.232	3.125	312	0.139	
7.142	3.392	3.75	375	0.11	
8.928	5	3.928	392	0.078	
10.714	6.607	4.107	410	0.062	
12.5	8.392	4.108	410	0.048	

 Table (5) The results of the adsorption capacity experiments for Iron-IIP2 and their relationship with the initial concentration

Ci	C <sub>f</sub>	C <sub>i</sub> -C <sub>f</sub>	O(umol/a)	$O(C_{\rm c}(\mathbf{L}/a))$	
(µmol/ml)	(µmol/ml)	(µmol/ml)	Q (µmor/g)	$\mathbf{Q}_{\mathbf{U}} \mathbf{C}_{\mathbf{f}} (\mathbf{L}_{\mathbf{f}} \mathbf{g})$	
0.178	0	0	0	0	
0.892	0	0	0	0	
1.785	0.446	1.34	134	0.3	
3.571	1.25	2.321	232	0.185	
5.357	2.5	2.857	285	0.114	
7.142	3.92	3.214	321	0.081	
8.928	5.35	3.571	357	0.066	
10.714	6.96	3.75	375	0.053	
12.5	8.75	3.75	375	0.042	



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NO.	Concentration Fe ion (µg/ml)	Fe ion found (µg/ml)	Recovery
1	1	0.98	98
2	5	4.93	98.6
3	10	9.95	99.5
4	20	19.93	99.6
5	30	29.8	99.3

#### Table (6) The results of the determination of Iron ion in synthetic organic solutions

Each extraction process considered results in a possible 20-fold increase in analyte concentration. It is a concentration of 5–25 ng/ml, which is easily measurable by GFAAS but not by FAAS, assuming we have a quantitative recovery.

Table (7) shows the typical performance information for the Iron-IIPs SPE and preconcentration method. The precision of the method for a standard, evaluated as the relative standard deviation (n = 8) was 3.5 ng/ml of Fe ions. The limit of detection, defined as the concentration of analyte that generates signals that are three times the blank's standard deviation plus the net blank's standard deviation ferocity for 100 ml of sample volume were 10 ng/ml.

 Table (7) Preconcentration procedure performance characteristics

Specification	Fe
Detection limit	10 ng/ml
precision	3.5
Linear rang	10 - 25 ng/ml
Equation of regression	$AA = 6.672 C_V - 0.0003$

#### **4.3.** Determination of Fe ion in Crude and fuel oil.

Samples of crude and fuel oil have been brought in from the refinery. After that, these solutions were added to the Iron-IIP-SPE packed column system in the same way as before. Table (8) shows the acquired findings as well as the recovery tests.



Samples	Iron (II) ion add (ppm)	Iron (II) ion found (ppm)	Recovery (%)
Crude oil 1	0.0	$6.8\pm0.5$	-
	10.00	$16.5 \pm 1.3$	98.2
	15.00	$21.6\pm0.9$	99
	20.0	$26.7\pm1.9$	99.6
Crude oil 2	0.0	$4.50\pm0.2$	-
	10.00	$14 \pm 1.1$	96.5
	15.00	$19 \pm 1.2$	97.4
	20.0	$24.6 \pm 1.5$	100.4
Fuel oil 3	0.0	$15 \pm 0.6$	-
	10.00	$24.7 \pm 1.3$	98.8
	15.00	$29.5\pm1.5$	98.3
	20.0	$34.8 \pm 1.2$	99.4

#### Table (8) Determination of Iron ion in Crude and fuel oil water

#### 5. Conclusion

Novel Iron-IIPs was prepared by bulk polymerization. Styrene and 1-vinyl imidazole selected as functional monomer and EGDMA as crosslinker. Besides, benzyl peroxide was used as an initiator in the presence of chloroform solvent. The optimal molar ratios of Iron ion to monomer and croslinker dosage were studied. The irregular shapes and three-dimension network structure of polymers it was possible to notice by SEM. The results of FT-IR and EDX proved the successful elution of Fe ions by **CH3OH/CH3COOH** (10:1 v/v) solution. Effects of operating time and initial Iron ion concentration on adsorption performance were investigated. Adsorption by IIPs was fast (adsorption equilibrium was reached within 10 min) and followed Langmuir isotherm models. The maximum adsorption capacity of the IIPs is (568) $\mu$ mol/g for Iron-IIP1 and (491)  $\mu$ mol/g for Iron -IIP2, which is higher than other sorbents reported in literatures. The elution procedure has very little effect on the cavity structure and chemical properties of the polymers, demonstrating that Iron -IIPs are highly stable and regenerable.



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