

Inductively Coupled Plasma-Atomic Emission Spectroscopy and Inductively Coupled Plasma-Mass Spectrometry Elemental Analysis of 65 Elements, 11 Oxides and Preparation of Catalyst in *Iris Postii* Mouterde.

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Abstract

Iris postii Mouterde, belonging to family Iridaceae is distributed in Kurdistan region/Iraq. The present study aimed to analyze the content of 65 elements (major and minor elements) Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Pd, Pr, Pt, Rb, Re, S, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn and Zr from roots and aerial parts of *Iris postii* by using inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma atomic emission spectroscopy (ICP-AES) techniques after microwave digestion and determination of 11 oxides SiO₂, Fe₂O₃, CaO, MgO, Na₂O, K₂O, Cr₂O₃, K₂O, TiO₂, MnO, P₂O₅, SrO and BaO by ICP-AES for the first time. Micromesoporous catalyst was hydrothermally synthesized by Tetra propyl ammonium hydroxide (TPAOH), Silicon dioxide (SiO₂) and sodium hydroxide (NaOH). Results show some elements, Iron (Fe), Barium (Ba), Manganese (Mn), Zinc (Zn), Strontium (Sr) and Boron (B) are present in the roots and aerial parts in a significant amount. Among them Fe has the highest concentration (340 ppm) and (1130 ppm) in the roots and aerial parts of *Iris postii* respectively, on the other hand, Ba (85.3

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ppm, 28.8 ppm), Mn (57.4 ppm, 19.4 ppm), Zn (29.2 ppm, 25.7 ppm), Sr (27.7 ppm, 16.95 ppm) and B (26 ppm, 14 ppm) are essential elements of the roots and aerial parts respectively.

Keywords: *Iris postii* Mouterde, Elemental analysis, ICP-AES and ICP-MS.

تحليل 65 عنصرا و 11 من أكاسيد وتحضير العوامل المحفزة من نبات (*Iris Postii*) بأستخدام التقنيتين البلازما المحتثة المقترن-الامتصاص الذرى الانبعائي و البلازما المحتثة المقترن- طيف الكتلة

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الخلاصة

ينتشر نبات *Iris postii* والذي يعود لعائلة *Iridacea* في إقليم كردستان العراق. الدراسة الحالية تتضمن تحليل مكونات 65 من العناصر ذات النسب العالية والقليلة (*Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Pd, Pr, Pt, Rb, Re, S, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn and Zr*) من جذور و أجزاء ورقية من نبات *Iris postii* وذلك بأستخدام التقنيتين (البلازما المحتثة المقترن-الامتصاص الذرى الانبعائي و البلازما المحتثة المقترن- طيف الكتلة). بعد ان تتم عملية الهضم في جهاز المايكروموجية، وكذلك تقدير 11 أكاسيد (*SiO₂, Fe₂O₃, CaO, MgO, Na₂O, K₂O, Cr₂O₃, K₂O, TiO₂, MnO, P₂O₅, SrO and BaO*) بواسطة تقنية ICP - AES لأول مرة. تم تحضير العامل المحفز مايكروميزوبورس بطريقة هيدروحرارية بواسطة رباعي بروبايل هيدروكساييد (TPAOH)، ثاني أوكسيد السليكون (SiO₂) وهيدروكسيد الصوديوم (NaOH). تظهر النتائج تواجد بعض العناصر (حديد، باريورم، منغنيز، زنك، الستروثيوم، بورون) في الجذور والأجزاء الورقية بكميات كبيرة، وخاصة عنصر الحديد له تركيز عالي (340ppm), (1130ppm) في جذور و أجزاء ورقية للنبات *Iris postii* على التوالي بينما في جانب أخر (*Ba (85.3 ppm, 28.8 ppm), Mn (57.4 ppm, 19.4 ppm), Zn (29.2 ppm, 25.7 ppm), Sr (27.7 ppm, 16.95 ppm) and B (26 ppm, 14 ppm)*) هي عناصر ضئيلة في الجذور وأجزاء الورقية على التوالي.

الكلمات المفتاحية: نبات *Iris postii*, تحليل المكونات. تقنية البلازما المحتثة المقترن-الامتصاص الذرى الانبعائي وتقنية البلازما المحتثة المقترن- طيف الكتلة.

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Introduction

Before scientific analysis developed, all medicinal plants were used by traditional beliefs on their properties in each country [1]. A large number of plants used in the traditional medicine are now part of the modern world health care system. Moreover, some plants are considered important not only as food but also for their therapeutic values, such as the ginger, green tea, walnuts and some others plants [2]. Many methods have been used for obtaining compounds for drug production; these include isolation from plants and other natural sources like microorganism, synthetic chemistry and combinatorial chemistry. There are challenges associated with each method, necessitating the need for one method to complement the other ones [3]. In the last few decades, the determination of minerals and trace elements are important to enhance production efficiency in plants and foods [4]. The human beings require both metallic and nonmetallic elements within certain permissible limits for growth and good health [5]. Determination and characterization the elemental composition of foods and related medical products is therefore very important for understanding their nutritive and medicinal value. These mineral elements may be broadly classified as macro (major) or micro (minor) elements based on their daily requirement [6]. Different techniques were used to determine the elemental contents of the medicinal plants from many parts of the world; such as, inductively coupled plasma optical emission spectrometry, Electrothermal AAS, Inductively coupled plasma mass spectrometry and Flame atomic absorption spectrometry [7]. ICP/AES and ICP-MS were benefits that high sensitivity and powerful multielement analysis capability. Furthermore, some common elements such as K, Na and P are essential for human health and important for nutritional purposes [8]. *Iris* belongs to the family of Iridaceae and an estimated 300 species [9, 10], most of them were used as a medication due to the different biological activities against cancer, inflammation, bacterial and viral infections, and other diseases [11, 12]. *Iris postii* is a medicinal plant belonging to the Iridaceae family and is widely distributed in Kurdistan region-Iraq and used by the local people as an anti-inflammation [13]. On the basis of the literature data, the elemental analysis of *Iris postii* have not yet been investigated. The aim of the present

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work is to do full and comprehensive analysis of the components of *Iris postii* using ICP-AES and ICP-MS.

Materials and Methods

1. Plant material

The aerial parts and roots of *Iris postii* Mouterde were collected in April 2016 from Korek Mountain in the Kurdistan region of Iraq. The plant parts were classified and identified by Dr. Abdullah Sardar from Salahaddin University-Erbil. A voucher specimen number 7230 was deposited at Education Salahaddin University Herbarium (ESUH)-Erbil, Kurdistan/Iraq. *Iris postii* (roots and aerial parts) were separately cleaned (washed with water) and air-dried under shade at room temperature (20-25°C) for 20 days. After drying, each plant part was finely powdered using a laboratory grinding mill, to provide homogeneous powder for the analysis. Powdered materials were stored in bottles and maintained at room temperature until analyses.

1. Determination of Elements

2.1 Sample Preparation

Preparation of vegetation samples for analysis was performed using ashing method in which, 100g sample is weighed into an ashing pan followed by controlling ignition at 475°C for 24 hours [14].

2.2 Sample preparation for analysis

1g of prepared dried plant sample is cold digested with nitric acid for 8 hours before being transferred to hot block for 15 minutes at 85°C followed by 2 hours at 115°C. The samples are subsequently cooled and brought up to volume with HCl. The resulting solution is mixed thoroughly and analyzed by ICP-MS and ICP-AES corrected for spectral interferences [14].

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3. Oxide determination

A prepared sample (0.100 g) is added to lithium metaborate/lithium tetraborate flux, mixed well and fused in a furnace at 1000°C. The resulting melt is then cooled and dissolved in 100 mL of 4% nitric acid/2% hydrochloric acid. This solution is then analyzed by ICP-AES and the results are corrected for spectral inter-element interferences. Oxide concentration is calculated from the determined elemental concentration and the result is reported in (Table 3).

Note: For samples that are high in sulphides, we may substitute a peroxide fusion in order to obtain better results.

4. Setting Parameters

RF incident Power (kW) 1.4, Plasma flow rate (l/minute) 15, Number of reading replicates 3, Auxiliary argon flow (l/minute) 1.0, Nebulizer flow rate (l/minute) 0.80, Pump flow rate (rpm) 15, Nebulizer Cyclonic type, Replicate read time (s) 10, Instrument stabilization delay (s) 30, Dwell time (s) 60, Sample uptake delay (s) 10. ALS Laboratory (Spain). Transmission electron microscopy (TEM) was recorded with a JEOL 2010 electron microscope operating at 200 kV. The sample was suspended in ethanol and dispersed on copper grid. Scanning electron microscopy (SEM) was recorded on a JSM-5410LV. The total surface area was calculated according to the BET isothermal equation and surface area were evaluated by t–plot method. The mesopore-size distribution was analyzed from desorption branch of the isotherm by the Barrett– Joyner–Halenda (BJH) method.

5. Preparation of catalyst

Silica oxide is added to an aqueous solution of tetra propyl ammonium hydroxide (TPAOH), sodium aluminate is dissolved in a concentrated sodium hydroxide (NaOH) solution and added to the first solution under vigorous stirring then the gel formation is transferred to an autoclave stainless steel and stirred at 160C for 48 hours until the crystallization occurs. The solid so obtained is filtered, washed several times with distilled water and dried overnight at 60C [14].

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Results and Discussions

The present study investigated a total of 65 elements, Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Pd, Pr, Pt, Rb, Re, S, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn and Zr in different concentrations by using ICP-AES/ICP-MS techniques from total plant body (root and shoot system) of *I. postii*. By using ICP-OES technique we could estimate a total of 8 elements such as Al, Ca, K, Mg, Na, P, S and Ti in percentage (%) level as illustrated in Table 1. While the most remain elements (minor elements) has been determined by ICP-MS in units of (ppm) as found in Table 2. However, 11 oxides SiO₂, Fe₂O₃, CaO, MgO, Na₂O, K₂O, Cr₂O₃, K₂O, TiO₂, MnO, P₂O₅, SrO and BaO were determined by ICP-AES as shown in table 3.

Table 1. Elemental composition of *Iris postii* (roots and aerial parts) by ICP-AES.

Elements	symbol	Root	A.P	Lower Limit (%)	Upper Limit (%)
Aluminum	Al	0.04	0.1	0.01	25
Calcium	Ca	0.87	1.59	0.01	40
Potassium	K	1.91	3.45	0.01	10
Magnesium	Mg	0.096	0.216	0.001	30
Sodium	Na	0.016	0.01	0.001	10
Phosphorus	P	0.336	0.417	0.001	5
Sulfur	S	0.15	0.3	0.01	10
Titanium	Ti	<0.001	0.001	0.001	10

A.P= Aerial part of *Iris postii*

Table 2. Elemental composition of *Iris postii* (roots and aerial parts) by ICP-MS.

Elements	symbol	LOD (ppm)	Root (ppm)	A.P (ppm)
Aurum	Au	0.0002	0.0002	0.0007
Silver	Ag	0.001	0.004	0.008
Arsenic	As	0.05	0.19	0.51
Boron	B	10	14	26
Barium	Ba	0.1	28.8	85.3
Beryllium	Be	0.01	0.02	0.04
Bismuth	Bi	0.001	0.004	0.007
Cadmium	Cd	0.002	0.144	0.157
Cerium	Ce	0.003	0.45	1.285
Cobalt	Co	0.002	0.35	0.841
Chromium	Cr	0.5	1.85	9.3

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Cesium	Cs	0.005	0.034	0.085
Copper	Cu	0.01	5.08	7
Dysprosium	Dy	0.005	0.036	0.106
Erbium	Er	0.003	0.018	0.049
Europium	Eu	0.003	0.011	0.032
Iron	Fe	0.001	340	1130
Gallium	Ga	0.01	0.112	0.31
Gadolinium	Gd	0.005	0.043	0.124
Germanium	Ge	0.005	0.012	0.022
Hafnium	Hf	0.002	0.004	0.011
Mercury	Hg	0.001	0.016	0.022
Holmium	Ho	0.001	0.007	0.02
Indium	In	0.005	<0.005	<0.005
Lanthanum	La	0.002	0.194	0.599
Lithium	Li	0.1	0.4	0.9
Lutetium	Lu	0.001	0.002	0.006
Manganese	Mn	1	19.4	57.4
Molybdenum	Mo	0.01	0.07	0.25
Niobium	Nb	0.002	0.016	0.07
Neodymium	Nd	0.001	0.23	0.615
Nickel	Ni	0.04	3.07	6.04
Praseodymium	Pr	0.003	0.053	0.149
Lead	Pb	0.01	0.21	0.62
Palladium	Pd	0.001	<0.001	<0.001
Platinum	Pt	0.001	<0.001	<0.001
Rubidium	Rb	0.01	3.17	4.44
Rhenium	Re	0.001	<0.001	<0.001
Antimony	Sb	0.02	0.02	0.05
Scandium	Sc	0.01	0.13	0.17
Selenium	Se	0.1	0.084	0.094
Samarium	Sm	0.003	0.046	0.125
Tin	Sn	0.01	0.02	0.03
Strontium	Sr	0.02	16.95	27.7
Tantalum	Ta	0.005	0.001	0.002
Terbium	Tb	0.001	0.006	0.019
Tellurium	Te	0.02	0.02	<0.02
Thorium	Th	0.002	0.016	0.042
Thallium	Tl	0.002	0.007	0.015
Thulium	Tm	0.001	0.002	0.007
Uranium	U	0.005	0.016	0.028
Vanadium	V	1	1.06	2.95
Tungsten	W	0.01	0.01	0.01
Yttrium	Y	0.003	0.2	0.523
Ytterbium	Yb	0.003	0.013	0.041
Zinc	Zn	0.1	25.7	29.2
Zirconium	Zr	0.02	0.17	0.46

LOD= Limit of detection by ppm, A.P= Aerial part of *Iris postii*

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Table 3. Oxide composition of *Iris postii* (roots and aerial parts) by ICP-AES.

Oxides	Symbol	Root %	A.P %
Silicon dioxide	SiO ₂	0.74	1.18
Aluminium oxide	Al ₂ O ₃	0.18	0.35
Iron(III) oxide	Fe ₂ O ₃	0.14	0.21
Calcium oxide	CaO	1.49	2.1
Magnesium oxide	MgO	0.44	0.38
Sodium oxide	Na ₂ O	0.04	0.04
Potassium oxide	K ₂ O	0.7	3.82
Chromium(III) oxide	Cr ₂ O ₃	<0.01	<0.01
Titanium dioxide	TiO ₂	0.01	0.02
Manganese(II) oxide	MnO	<0.01	0.01
Titanium dioxide	P ₂ O ₅	0.19	0.88
Strontium oxide	SrO	<0.01	<0.01
Barium oxide	BaO	<0.01	0.01

Units: %, Lower limit= 0.01, Upper limit= 100

The aerial part of the plant body contains higher level of most of the estimated elements than the root part. As a result, each of Iron (Fe), Barium (Ba), Manganese (Mn), Zinc (Zn), Strontium (Sr) and Barium (B) are present in the roots and aerial parts in significant amount. Among these elements, we found that, Fe is the highest concentration (340 ppm) and (1130 ppm) in the roots and aerial parts of *Iris postii* respectively, while, each of Ba (85.3 ppm, 28.8 ppm), Mn (57.4 ppm, 19.4 ppm), Zn (29.2 ppm, 25.7 ppm), Sr (27.7 ppm, 16.95 ppm), B (26 ppm, 14 ppm), Cr (1.85 ppm, 9.3 ppm), Cu (5.08 ppm, 7 ppm) and Ni (3.07 ppm, 6.04 ppm) are in the second level of elements that found in roots and aerial parts respectively. At the same time, the Au (0.0002 ppm and 0.0007 ppm), was recorded as a minimum concentrations from roots and aerial parts respectively. The results showed that both aerial part and root samples contained many essential elements (Al, Ca, Co, Cr, Fe, Mg, Mn, Mo, Ni, P, Se, Sr, V and Zn) which demand by our body. Besides the wholesome elements, the contents of heavy metals (As, Cu, Cd, Hg and Pb) are also necessary to identify the quality of a plant.

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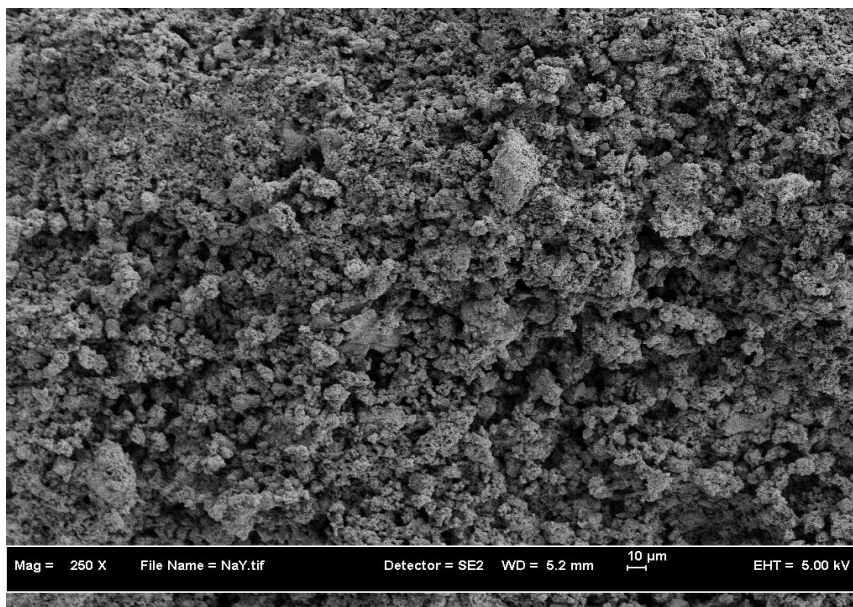


Fig. 1 SEM image for solid catalyst

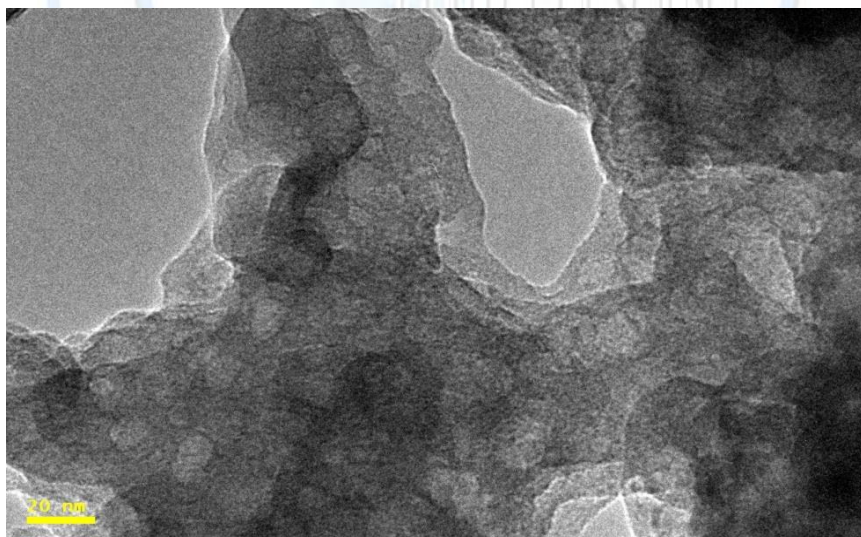


Fig.2 TEM image for solid catalyst

Figures 1 and 2 show typical SEM and TEM images of calcined Catalyst. Some catalyst nanoparticles were enwrapped. The TEM images indicate well-ordered hexagonal arrays of

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mesopores structure. Simultaneously, the sample catalyst contains some smaller micro particles and rough surfaces of particles cause the pore network and interconnect with the mesopores and micropores. The BET surface area of the mesoporous catalyst is about 89 m²/g, consisting of a micro pore area of 29.5 m²/g. The mesoporous catalyst can conquer pore size limitation of catalyst and makes their pore size distributed from micro scale to meso scale.

Conclusions

The present study estimated the content of 65 elements and 11 oxides of aerial parts and roots of *Iris postii* Mouterde which collected from Korek Mountain in Kurdistan region/Iraq by using the ICP-AES/ ICP-MS technique for the first time. The ICP-MS/ICP-AES show to be powerful and sensitive techniques for the determination of macro (major) or micro (minor) elements in the herbal plant *Iris postii* Mouterde. By comparing the total contents of the total of 65 elements and 11 oxides, as shown in (Table 1, Table 2 and Table 3), we found that the distribution patterns of different elements in the aerial parts and roots of *Iris postii*, were different. The elements present in the medicinal plant *Iris postii* (arial parts and roots) play an important role in the treatment of different diseases.

Acknowledgements

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References

1. Maryam, K.; Bouzari, Z.; Zahra, B.; Mehrdad, K.; Mahtab, Z. *BMC Res. Notes*. 2012, 133(5), 135-139.
2. Saklani, A.; Kutty, S. *Drug Discov. Today*. 2008, 13(3-4), 161-71.
3. Balunas, M.; Kinghorn, A. *Life scie*. 2005, 78(5), 431-441.
4. Rodriguez, L.; Morales, D.; Rodriguez, E.; Romero, C. *J. of Food Comp. and Anal*. 2011, 24, 1081- 1090.

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5. Djingova, R.; Kovacheva, P.; Wagner, G.; Markert, B. *Sci. Total Environ.* 2003, 308, 235-246.
6. Zbinden, P.; Andrey, D. *Atom Spectrosc.* 1998, 19(6), 214-219.
7. Rodushkin, I.; Ruth, I.; Huhtasaari, A. *Anal. Chim. Acta.* 1999, 378(1-3), 191-200.
8. Korkmaz, K.; Kara, S.; Ozkutlu, F.; Gul, F. *African J. of Agricul. Rese.* 2010, 5, 463-467.
9. Muhammad, K.; Zaheer-ud-din, A.; Muhammad, Z.; Sabahat Z. *Biologica (Pakistan)*. 2013, 59(2), 345-348.
10. Sabrin, R.; Gamal, A.; Nawal, M. *Molecules.* 2012, 17, 2587-2598.
11. Farag, S.; Kimura, Y.; Ito, Z.; Takayasu, J.; Tokuda, H.; Hatano, T. *J. Nat. Med.* 2009, 63, 91-95.
12. Guo-Yong, X.; Xiao-Ying, Q.; Rui, L.; Qi, W.; Bin-Bin, L.; Guo-Kai, W.; Guang-Kai, X.; Rui, W.; Min-Jian, *Natu. Prod. Res.* 2013, 10, 1-5.
13. Townsend, C.; Guest, E. *Flora of Iraq*; 8th Eds.; Ministry of Agriculture & Agrarian Reform; Baghdad: Iraq, 1985.
14. Gozzini, D. 2012. Ph.D. Thesis, IUSS&Istituto Universitario di Studi Superiori – Pavia, Italy.