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Analysis of major and trace elements for the residual manufactures of Taif rose water by dual view inductively coupled plasma optical emission spectrometry (ICP-OES)

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ABSTRACT

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) is ideally suited for the analysis of elements in water and wastewaters. The contamination levels in urban and industrial effluent such as the effluent rose water need to be specified and controlled as a waste management strategy for this kind of effluent, by recycling or reused of the effluent of the rose water in several industries such as applied to cosmetics and agriculture depending on the kinds of the elements found in effluent rose water. The aim of this study is to evaluate and characterize the elements (qualitative and quantitative analysis) of the effluent rose water manufactory in Al Hada- Taif, which are widely used in different parts of Taif. The element concentration of the effluents rose water will be determined using ICP-OES techniques. Samples was collected overall Al hada. To our knowledge there has been no research or work investigated on the effluent of the manufactures of hydro- distillation of the Taif rose. The results showed this method suitable for the analysis of nineteen elements and their associated wavelengths for different kinds of samples.

Key words: Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), Taif, elements, rose water.

INTRODUCTION

The inductively coupled plasma optical emission spectrometry (ICP-OES) is widely used for the analysis of elements in all kinds of water (drinking water, wastewater and effluent water from industry) [1]. Because of its multi-elements determination capability, its high dynamic liner range and its sensitivity. Levels of trace, minor and major elements can be determiner simultaneously, thus ensuring low analysis cost [2].

Rose essential oil, is highly prized product used in perfumery, cosmetic and pharmacy [3&4]. Bulgaria and turkey are the main rose processing countries in the world which extract the rose oil by water-steam distillation of Rosa damascene Mill, petals. Since more than 3,000 Kg of petals yield 1 Kg of rose oil and 1 Kg of fresh raw material gives approximately 2 Kg of residue on a wet weight basis, several thousand tons of waste material annually result from the distilleries in Bulgaria alone. Due to the selective rose oil recovery, using hydro-distillation extraction (the rose petals mixed with water in a copper pot and heat gently, the distillation process is done slowly and the majority of the essential oil are obtained by steam condensation of the product then collected and separated), the elements compounds are retained in the waste water effluent of the process.

DimitarHristozov [5] used ICP-OES to analysis eight metals (Cd, Co, Cr, Cu, Mn, Ni, Pb and Zn) in sewage sludge. Microwave digestion was used for comparison and multivariate approach for optimization. Luis et al. [6&7]employed on line per concentration procedure using solid phase extraction for the determination of copper in different water samples by ICP-OES. The methodology was successfully applied to the determination of copper and zinc in tap, mineral, river water samples. Vimlesh Chand [8] assessed heavy metals such as (Al, As, Co, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) in sediment using ICP-OES. Sewage sludge, compost from solid waste and effluent from industries often have relatively high elements concentrations such organic and semi- organic waste are often applied in agriculture as fertilizers, and although the recycling of waste is desirable, waste contamination by toxic element such as mercury may lead to higher concentration of mercury in the soil and environment which it is applied, compared to those found when the soil is conventionally fertilized [9&10]. Christine and Doug [11] evaluated microwave digestion to prepare fertilizer for determination of As, Ca, Cd, Cu, Cr, Fe, K, Na, Mg, Mn, P, Pb, Se and Zn by simultaneous ICP-OES. The main concern with water recycling is the uncertainty of its quality and the potential to health problems and cause environmental harm, that need for better water management. One of the methods of managing water efficiently is to recycle or reuse water, which can reduce the pressure on both the supply and disposal aspects of water management. Atomic spectrometry technique were popular tool, to the determination of elements in deferent types of sample described by Salman[12-14]

Therefore, the aim of the present study is to identified and determine the elements in distilled rose petals and cook water employing ICP-OES with dual view to give a full characterization of the effluent.

MATERIALS AND METHODS

2.1 Inductively Coupled plasma-Optical Emission Spectrometry (ICP-OES) instrument:

The measurements were performed using PerkinElmer Optima 2100 DV ICP-OES instrument (PerkinElmer, Inc. Shelton, Ct, USA) equipped with WinLab32 for ICP Version 4.0 software for simultaneous measurement of all analytic wavelengths of interest as shown in Table(1). The Optima 2100 DV has been optimized to provide high speed analysis. By combining an SCD detector and an echelle optical system, the Optima 2100 DV can measure all elements simultaneously. Its wavelength flexibility allows the end users the end to easily add new elements as their program needs change. The ICP torch is physically mounted in an axial orientation in the instrument shielded torch box, but can be viewed either axially or radially. The view mode is user-selectable on an element-by-element basis. A shear flow (compressed air) provides elimination of the cool plasma tail to allow direct observation of the plasma normal analytical zone, thus minimizing chemical matrix effects when the axial-view mode is employed.

Table (1): List of wavelengths used and Plasma View Selected with analysis of instrument calibrated solution

Element	Wavelength (nm)	Calibration up to (mg/L)	Plasma View
Arsenic	193.696	10.08	Axial
Beryllium	234.861	10.11	Radial
Calcium	317.933	10.21	Radial
Cadmium	214.440	10.15	Axial
Chromium	205.560	9.96	Radial
Copper	224.700	9.93	Radial
Iron	239.562	10.25	Radial
Lithium	610.362	9.95	Axial
Magnesium	280.271	10.17	Radial
Manganese	257.610	10.18	Radial
Molybdenum	202.031	9.76	Axial
Nickel	232.003	10.11	Radial
Lead	220.353	10.02	Axial
Antimony	206.836	10.02	Axial
Selenium	206.279	9.82	Axial
Titanium	336.121	9.96	Radial
Thallium	351.924	9.89	Axial
Vanadium	310.230	9.89	Radial
Zinc	202.548	9.90	Axial

The sample introduction unit consisted of a cyclonic spray chamber and a low flow Meinhard concentric glass nebulizer. The cyclonic spray chamber ensures high sample transfer to the plasma and fast rinse in and out times, which improves productivity. The low flow Meinhard nebulizer permits lower nebulizer gas flow rate, useful for spectral lines with high excitation energies and for providing more robust plasma, which is essential for waste water samples.

The read time was set for a variable range from 2 s to 5 s using the auto-integration mode. Table (2) shows the optimization detailed for the instrument parameters. The PerkinElmer S10 Auto-sampler was used for high throughput and automated analysis. The auto-sampler automates standard and sample introduction for instrument calibration and sample analysis.

Table (2): Optima 2100 DV ICP-OES instrumental conditions and experimental parameters

Plasma gas flow	15 L/min
Auxiliary gas flow	0.2 L/min
Nebulizer gas flow	0.6 L/min
RF power	1450 watts
Peristaltic pump flow rate	1.0 ml/ min
Spray chamber	Cyclonic
Nebulizer	low flow meinhard
Injector	Alumina, 2.0 mm i.d
Sample tubing	Standard 0.76 mm i.d
Drain tubing	Standard 1.14 mm i.d
Sample capillary	Teflon 1 mm i.d
Resolution	Normal
Processing mode	Peak area
Torch Orientation	Axial
Auto Integration (min-max)	2-5 sec
Sample Vials	Polypropylene

2.2 Reagents and standards

PerkinElmer high purity standards for ICP were used as the stock standards for preparing working standards. A total of nineteen elements were used for qualitative monitoring and quantitative calibration as installed in (Table 1). Standards solution were prepared using 1000 µg/ml standards of each metal was quantitatively (1 ml) transferred to a 100 ml volumetric flask and made up to the mark with 5% H₂NO₃ and stored.

2.3 Distillation procedure

Rose-picking in the Rose Valley and mountains near the town of Al-hada, Taif, KSA during the early, virtually all rose oil was distilled on-site using direct-fire stills operated by the farmers. Modern stills are made of copper and are heated with an open propane gas fire from below the apparatus would be set up. Although this sounds rather primitive, the yield produced from this type of amounts to 1 kilo of oil for every 3,800 to 4,000 kilos of roses petals. Amazingly, this is a considerably higher figure than can be achieved by modern industrial distillation techniques. The roses can be distilled in the usual way by directly heating, because the petals compact to form a large mass that the heat can penetrate.

During distillation an amount of oil is absorbed into the distillation water, and this is known as the 'First Water' distillate water. The rose oil must be recovered from this water to produce an acceptable yield, and this is achieved by re-distilling the water to separate the oil; a process known as cohobating. The 'Second Water' remaining after the process of cohobating is then sold as rose hydrosol (Alaros water) or re-cycled in the still for the next batch of flowers.

The batch in one pot equal to 70 kilos of the rose damascene with 100 liter water. The still is fired for 3-4 hours with gentle heating.

2.4 Sample Preparation

Sample preparation is the critical step in any analysis, especially at trace levels. Contamination during the preparation step or loss of analytes can dramatically affect the accuracy of results. The ability to digest difficult samples reproducibly or digest in a closed environment can be especially important as detection limit requirements

continue to be driven to lower levels. All samples and standards were prepared using prepared multi-element stock solutions. The calibration was performed using a 10 ppm (final concentration) multi-element stock solution for each elements under investigate as shown in table (1). Deionized use in the preparation of reagents and in the dilution of samples. . Micropipettes (Eppendorf®, Germany) with disposable tips were used for pipetting all solutions.

Several samples (cook water) collected from the manufacturing process using the hydro- distillation process plants to extract the essential oil from Al Hada- Al Taif province, KSA in spring 2014 from different production- batches, and one sample prepared in laboratory in the same miner of the factory. The fresh petals underwent the process of extraction with water. The composition of the mixture obtained after extraction for about four hours it is completed, the major and trace elements of the cook water was determined with the ICP-OES. The effluent (cook water) analyzed in order to collect date on the elemental solubility's in solutions considered to be commonly extraction solutions and to determine the level. The suspension solution samples were passed through a stainless steel mesh sieves before filtering by Whatman filter paper using Buchner funnel. The sample Centrifuge and stored in refrigerator. Aliquot of the samples was diluted with purified water to bring the concentration of the samples within the range of the calibration curves. All samplestoanalysiswerepassedthroughthe0.45um desk filter to remove the very fine appreciate that lead to clogged the nebulization system of the ICP-OES.

RESULTS AND DISCUSSION

Hydro-distillation is the most favored method of production of Taif essential oil and rose water from the Taif rose petals in Al-Hadaregion , a wide variety of consumer good such as cosmetics, perfumes, pharmaceutical as well as the production for second water used in food flavours and in cosmetics it is also added to tea, ice cream, cookies and other sweets in small quantities so the specification of the water used in these industry it is important.

Results for the determination of elements in tap water and the second water with optimum conditions for ICP-OES demonstrated in table (3) as well as List of wavelengths and calibration standards of the analyte. Table (3) also indicated the concentration of a major elements (Ca and Mg) and trace elements (As, Be, Cd, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Se, Ti, Tl, V, Zn) were determined using an inductively coupled plasma optical emission spectrometry calibrated with mixture of each elements under study. The results shows that the level of the elements in the regulars region.

Table (3): Results for the determination of elements in tap water and the secondwater in Rose petals process in Al-Hada factory.

Elements	Wavelength (nm)	Tap water (mg/l) X 10 ⁻²	Second water (mg/l) X 10 ⁻²
Arsenic	193.696	<LOD	<LOD
Beryllium	234.861	0.3	1.2
Calcium	317.933	3364.0	216.2
Cadmium	214.440	0.5	2.1
Chromium	205.560	0.3	1.6
Copper	224.700	1.1	2.1
Iron	239.562	0.3	2.2
Lithium	610.362	<LOD	14.7
Magnesium	280.271	621.6	35.6
Manganese	257.610	0.4	1.4
Molybdenum	202.031	1.0	<LOD
Nickel	232.003	0.6	0.6
Lead	220.353	0.4	4.2
Antimony	206.836	0.9	3.0
Selenium	206.279	<LOD	<LOD
Titanium	336.121	<LOD	0.6
Thallium	351.924	<LOD	<LOD
Vanadium	310.230	9.4	18.2
Zinc	202.548	<LOD	01.0

The LODs were for all elements < 1 ng/ml[15]

The detection power of the ICP-OES 1450 Watts was sufficient for the determination of elements under study. The relative standard deviation (RSD) were approximately 2% for the measurements performed by ICP-OES were

calculated as the concentration equivalent to three times the standards deviation (3σ) of the signal of the blank solution.

Two samples of effluents were analyzed in order to collect data on elementals solubilities in water obtained after four hours extraction considered to be commonly used extraction solution and to determine the level of elements in the same manner. The results showed there were four major elements such as (Ca, Cu, Mg, Fe) of a range of milligram per liter 70.05, 15.21, 1.51, 1.53 respectively. The analysis parameters for the rest of the elements showed low level not far from the concentration of these elements in the analysis of second water. So there is no required to make extra precaution when handling these effluent and it can handle with a logical fashion.

CONCLUSION

In conclusion, it is clear that ICP-OES is a strong contender in the determination of several elements in the water and wastewater of the hydro-distillation of the rose petals that can say it is very helpful technique to protect the environment and rural environment surrounding the manufacture area by knowing the constituent concentration of the effluent if the manufacture thought the wastewater in the area.

The driving force of this study was the need for a study of method of analyzing element in the waters thought the use of ICP-OES. This technique has come into its own in recent years due to its speed, sensitivity and multi-elements capabilities, as an analytical method for the determination of many elements in a wide variety of matrices. It is benefits have rendered it the mainstay of many analytical laboratories in the environmental, health and general industrial areas. Currently, the distilled first water sell in glass container 250 ml, and for the effluent water (cook water) same factory though it in the sounding area and same stored in cooled place to reused (recycle) in another batch or store it for the next session. Present study, the level of nineteen elements was tested in the studied ash first water as well as in cook water. The concentration of many elements was very low and therefore could be detected only with the use of the very sensitive methods of ICP-OES.

Fortunately all the sample analysis it shows that the centration of all element and the waters analysis it were free from the heavy element such as V, Mo and free from poison elements. At the same time, the level of Arsenic in very low and fare from the detection by the instruments. Advantage of the ICP-OES in this study was the relative freedom it allowed from matrix effects. This meant that the different extraction methods could not toused. Both the lines used were free from interference.

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