

Determination of essential and trace elements of red fruits and leaves of *Pistacia lentiscus* L. plant from western Algeria using k_0 -INAA technique

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Abstract— The present study describes the elemental composition of essential and traces contents of two organs of *Pistacia lentiscus* L. plants. The samples were collected from plants growing wild in the Tiaret region of Western Algeria. In this work, we focus to analyze red fruits and leaves extracted from the plants by k_0 -based on neutron activation analysis technique in our laboratory, the samples were prepared including drying, crushing, sifting and homogenizing before the irradiation step. The samples were introduced in the pure aluminum capsule for irradiation during 3 hours in the experimental channel at Es-Salam research reactor. In the irradiation position, the neutron flux parameters $\alpha = 0.027$, $f = 28.8$ and $\Phi_{th} = 3.7610^{13}$ (n/cm².s) were determined experimentally by several techniques. Twenty one elements were measured for both samples. Essential elements such as Ca, K, Fe, Na and Zn were found relatively higher in the red fruits than in the leaves except for calcium and iron. In the other hand, almost of the concentration values of trace elements are comparable in red fruits and leaves. For this study, the concept (QC/QA) is considered to evaluate the accuracy of the method. It was established by analyzing certified reference materials representing leaves matrix. Two CRMs NIST 1573a tomato leaves and GSV4 tea leaves were analyzed simultaneously with the samples. Three statistical parameters Z-score, U-score and bias were determined and discussed.

Index Terms— Essential and trace elements, k_0 -INAA, *Pistacia lentiscus* L, Red fruits and leaves.

I. INTRODUCTION

During fifteen years, the effective research activities of the neutron activation analysis department at Es-Salam research Reactor were based in several fields of life using soils, plants, nutrients, materials, human and animal tissues matrices. The analytical tools such as relative and k_0 -NAA techniques have been used for all analysis [01-05]. The Chapter: Concepts, Instrumentation and Techniques of Neutron Activation Analysis [06], an overview of the most recent applications of the INAA and k_0 -NAA techniques applied in our laboratory. Examples of such samples, within a selected group of disciplines are milk, milk formulae and salt (nutrition),

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human hair and medicinal seeds (biomedicine), cigarette tobacco (environmental and health related fields) and iron ores (exploration and mining).

This work aim to get elemental concentrations of *Pistacia lentiscus*.L plants (Fruits and leaves) by nuclear analytical techniques based on neutron activation analysis k_0 -INAA used in our laboratory. Many advantages of *Pistacia lentiscus*.L can be listed as: antiatherogenic, antimicrobial and antimutagenic, antioxidant, antifungal, lipid lowering, hepatoprotective, anticancer, anthelmintic, wound healing, hypotensive, antiarthritic, antigout activity and also in the treatment of functional dyspepsia. It is rich in calcium and phosphorus Because of high water content (78–82%, w/w) [08]. The mean objective of this work is to give some information about the composition differences of studied plants by the determination of essential and toxic elements of red fruits and leaves of *Pistacia lentiscus* L.

Different techniques were applied to determine the elemental content in the medicinal plant materials and their infusions, e.g., neutron activation analysis (NAA)[09], inductively coupled plasma atomic emission or mass spectrometry (ICP-AES, ICP-MS) [10], atomic absorption spectrometry (AAS) [11,12,13], and total reflection X-ray fluorescence spectrometry (TXRF) [14,15]. In addition, energy dispersive X-ray fluorescence (ED-XRF) with a combination of different radionuclide sources showed a reliable determination of some elements such as Br, Ca, Fe, K, Mn, Rb, Sr, and Zn in plant bioindicators[16].

This paper treats the analytical determinations of essential and trace elements of red fruits and leaves of *Pistacia lentiscus* L leaves and compared the results with reported literature data. The elemental contents have been determined at different levels major, minor and trace by k_0 -NAA method.

II. EXPERIMENTAL

A. Sampling and sample preparation

Samples of *Pistacia lentiscus* .L were collected in Western Algeria (Forest Tiaret, 268 km Southwest of Algiers) in February 2013 *Pistacia lentiscus* L, cCommonly known as mastic tree or mastagi, it is found in the flora of many Mediterranean regions. Fruits and Leaves were separated from the stems and soaked in water to remove any dirt. Further, its surface contamination was wiped with tissue paper and left for natural air drying during 20 days.

The samples were powdered in agate mortar and passed through 200-mesh sieve. Various Certified Reference Materials such as Chinese tea leaves GSV4 and Tomato leaves NIST-1573a were also dried as per recommended procedure before use. Moisture content was determined by

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heating the leaves at 104°C for 4 hours and found to be 82%. According to the k_0 -methodology, the use of flux monitors is required due to the specific calculation based on the comparator factor F_c of gold [27-31]. Triplicate samples of *Pistacia lentiscus* L. leaves red fruit were prepared. About one hundred thirteen milligrams aliquots each of powdered samples and CRMs were accurately weighed and packed in aluminum foil for long irradiation. About 7 mg of 0.1%-Al alloy wire provided by IRMM (1 mm diameter and 3 mm length) were also weighed and sandwiched between sample and CRM. In this stage, all prepared samples/CRMs/monitors were introduced in the pure cylindrical aluminum capsule for irradiation.

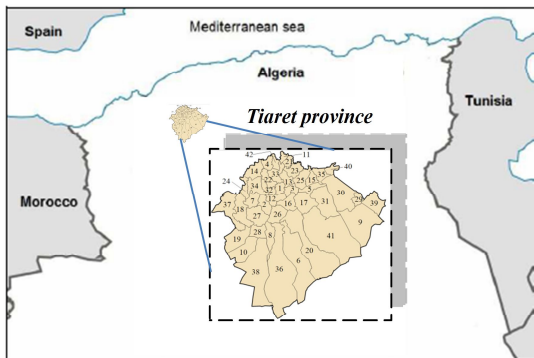


Fig 1: Map of investigated area in Tiaret province

B. Irradiation at Es-Salam research reactor

The prepared capsule was placed in the appropriate irradiation site during 4 hours at 3.761013n.cm-2.s-1 of Es-Salam research reactor. This channel is characterized by two parameters using different methods with and without cadmium where $\alpha = 0.027$, $f = 28.8$ [17].

C. Measurement and treatment of gamma rays spectra

All activities were measured using a coaxial HPGe detector (Canberra) with 1.8 keV resolutions at 1332 keV of ^{60}Co with 40% relative efficiency with GENIE-2k software (Canberra) [18]. Cooling time was in the range of 2 days and the counting about 1000 seconds. The typical gamma rays spectra of red fruits and leaves of *Pistacia lentiscus* L are presented in the figure 2. The second measurements were executed after 12 - 16 days. The details about the concept and the mathematical developments of the k_0 -NAA and relative methods have been demonstrated by in many research papers [17-19]. All spectra are deconvoluted with the commercial software HyperLab 2005 [20] with resulting to files peak table (*.PTF) and a spectrum (*.SPE) which are used as input for KayWare. This is a specific software package developed for k_0 -based NAA at DSM Resolve in the 1990s and at present commercialized by INW Gent and k_0 -Ware in the Netherlands [21].

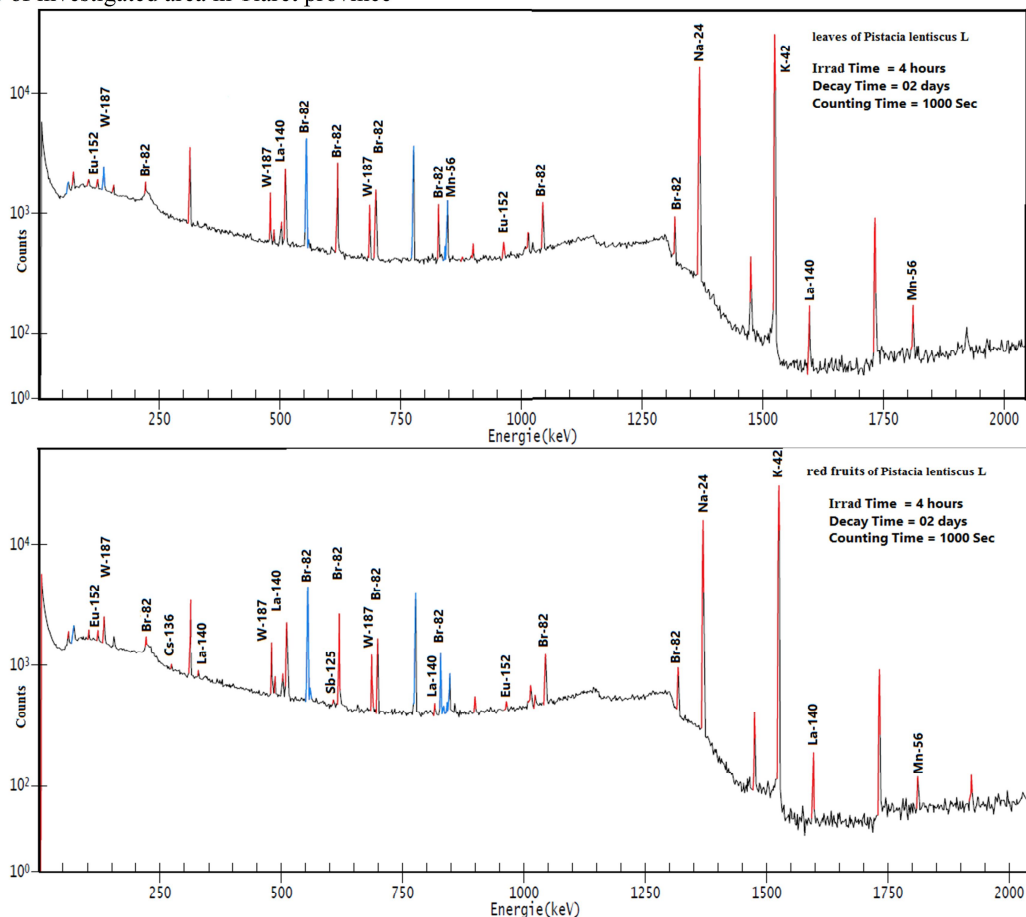


Fig2. Typical gamma ray spectra collected 2 days after irradiation during 1000 sec of red fruits and leaves of *Pistacia lentiscus* L

III. METHOD AND CALCULATIONS

Use of the k_0 -method in reactor neutron activation analysis is based on irradiation of Au wire and Zr foil together with the intended sample in order to determine the thermal to epithermal neutron flux ratio f and the parameter α , allowing the deviation of the epithermal neutron flux distribution from the $1/E$ law [22]. The concentration of an element can be determined as:

$$\rho_x (\text{ppm}) = \frac{\left[\frac{N_p/t_m}{\text{SDCW}} \right]_x}{\left[\frac{N_p/t_m}{\text{SDCW}} \right]_{\text{Au}}} \cdot \frac{1}{k_{0,\text{Au}(x)}} \cdot \frac{G_{h,\text{Au}} f + G_{e,\text{Au}} Q_{0,\text{Au}} (\alpha\alpha)}{G_{h,x} f + G_{e,x} Q_{0,x} (\alpha\alpha)} \cdot \frac{\epsilon_{p,\text{Au}}}{\epsilon_{p,x}} \times 10^6 \quad (1)$$

where the indices x and Au refer to the sample and the monitor, respectively; W_{Au} and W_x represent the mass of the gold monitor and the sample (in g); N_p is the measured peak area, corrected for dead time and true coincidence; S , D , C are the saturation, decay and counting factors, respectively;

IV. QUALITY CONTROL AND QUALITY ASSURANCE

For k_0 -NAA determinations, two standards NIST1573a and GSV 4 were used for quality control purposes. In the case of k_0 -NAA the accuracy depend on the data of experimental determination of neutron flux parameters and efficiency calibration of HPGe detector. As the method also offers some intrinsic quality control aspects, the uncertainties obtained

t_m is the measuring time; G_{th} and G_e are the correction factors for thermal and epithermal neutron self-shielding, respectively; f is the thermal to epithermal neutron flux ratio; $Q_0(\alpha)$ is obtained from, where $Q_0 = I_0/\sigma_0$ is the resonance energy, α is the experimentally measured factor for the deviation of the epithermal neutron flux distribution from the $1/E$ law and ϵ_p is the full energy peak efficiency.

The k_0 -factors, which are independent at irradiation and measurement conditions, are tabulated and published in literature as generally useful nuclear parameters. According to the HøGDAHL formalism, the k_0 -NAA technique uses input parameters, such as (1) the epithermal neutron flux shape factor (α), (2) the subcadmium-to-epithermal neutron flux ratio (f), (3) the full energy peak detection (ϵ_p), and nuclear data on Q_0 (ratio of resonance integral I_0 to thermal neutron cross-section σ_0) and k_0 . The computational and experimental procedures were created to simplify the determination of α and f . The relevant data for the nuclides used in k_0 -NAA method were presented in table 1.

are sufficient to perform all the necessary measurements on the reference materials. For QC/QA of the techniques, the comparative study is made between INAA and k_0 -NAA. In the other hand, the validation of results obtained by both techniques is carried out directly by “determined/certified ratios” in NIST1573a and GSV4 measured by both techniques [23].

Table1: Relevant data for the nuclides used in k_0 -NAA [25]

Element	Radio nucleide	Half life	Energy (keV)	k_0	Element	Radio nucleide	Half life	Energy (keV)	k_0
As	As 76	26.24 h	559.1 657.1	0.0483 (1.6) 0.00661	La	La 140	40.27 h	487.02 815.8 1596.21	0.0637 (0.9) 0.0332 (0.6) 0.134 (1.1)
Ba	Ba 131	11.50 d	496.3	0.0000648 (1.4)	Mn	Mn56	2.579 h	846.77 1810.7	0.496 (0.6) 0.135 (0.4)
Br	Br 82m	35.30 h	554.3 698.08 776.5	0.0238 (1.1) 0.00938 (0.9) 0.00938 (0.8)	Na	Na 24	14.95 h	1368.6	0.0468 (0.6)
Ca	Ca 47	4.536 d	1297.1	0.0000000954 (1.7)	Rb	Rb 86	18.631 d	1077	0.000765 (1)
Ce	Ce 141	32.508 d	145.4	0.00366 (0.9)	Sb	Sb 124	60.20 d	1691	0.0141 (1.1)
Co	Co 60	5.2714 y	1173.2 1332.5	1.32 (0.4) 1.32 (0.4)	Sc	S c 46	83.83 d	889.3 1120.5	1.22 (0.4) 1.22 (1.1)
Cr	Cr 51	27.7025 d	320.1	0.00262 (0.5)	Sm	Sm 153	46.50 h	103.2	0.231 (0.4)
Cs	Cs 134	2.0648 y	604.7 795.8 121.78	0.476 (2) 0.415 (2) 12.8 (0.8)	Sr	Sr 85	64.84 d	514	0.0000915 (0.9)
Eu	Eu 152	13.537 y	344.28 1408.01	11.9 (0.9) 9.36 (0.6)	Th	Pa 233	26.96 d	300.1 311.9	0.00437 (0.3) 0.0252 (0.5)
Fe	Fe 59	44.5 d	1099.3 1291.6 133	0.0000777 (0.4) 0.0000593 (0.4) 0.0237 (0.6)	W	W187	23.72 h	479.53 685.77 113.8	0.0297 (1) 0.0371 (0.5) 0.00942 (1.3)
Hf	Hf 181	42.39 d	345.9 482.2	0.00793 0.0456 (0.9)	Yb	Yb157	4.185 d	282.5 396.3	0.0146 (0.3) 0.0312(0.6)
K	K 42	12.360 h	312.76 ; 1524.7	0.000746 0.000946 (0.6)	Zn	Zn 65	244.26 d	1115.5	0.00572 (0.4)

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The objective was to assess the quality of our laboratory applying k₀-NAA. The statistical evaluation was carried out for determining analyses performance and the meaning of the results. Two statistics such as Z-score and U-score are most often used. The evaluation using U-score in clues uncertainties of the measurements and the uncertainty the assigned value. On the other hand, in the case of Z-score the uncertainty of the measurement is no taken account for the evaluation of performance. However, the performance of a method is more significant by applying U-score. In this study, the following questions were used in the calculations:

$$U_{score} = \frac{|X_{Lab} - X_{Ref}|}{\sqrt{\mu_{Lab}^2 + \sigma_{Ref}^2}}$$

Where: x_{Lab}, μ_{Lab}, x_{Ref} and σ_{Ref} are the laboratory results, standard deviation, the recommended and standards uncertainties, respectively. The laboratory performance is evaluated as satisfactory if U_{score} ≤ 1 and unsatisfactory for U_{score} > 1.

$$Z_{score} = \frac{X_{lab} - X_{Ref}}{\mu_{Ref}}$$

Where the laboratory performance is evaluated as satisfactory if Z_{score} ≤ 2, questionable for 2 < Z_{score} < 3 and unsatisfactory for Z_{score} ≥ 3 [24].

V. RESULTS AND DISCUSSIONS

Elemental concentrations were calculated by using CRMs as comparators for the k₀-NAA method. The concentration values obtained in this work for CRMs NIST1573a and

GSV4 with their certified/information values were presented in the table 2. A comparison of concentration of red fruits and leaves of Pistacia lentiscus L. plant samples are given in Table 3. In addition, two sets of data have been compared with our results reference presented also in Table 2. It is observed that our data in Table 2 are good agreement with certified values.

The variation in elemental concentration is mainly attributed to the differences in botanical structure, as well as in the mineral composition of the soil in which the plants are cultivated. Other factors responsible for a variation in elemental content are preferential absorbability of the plant, use of fertilizers, irrigation water and climatological conditions [32]. In this study, twenty four elements were analyzed in both samples of Pistacia lentiscus L plants representing the major : K, Ca, and the minor and trace: As, Ba, Br, Ce, Co, Cr, Cs, Eu, Mn, Fe, Hf, La, Na, Rb, Sb, Sc Sm, Sr, W, Th, Yb and Zn. It can be seen that the composition of major, minor and trace elements are comparable in two types of Pistacia lentiscus L except the value of iron and calcium determined in leaves of P.L sample that is higher than in red fruits of P.L. The same observation is made for Ba, Br, Co, Na, Mn, La, Sm and Zn for which the ratio of leaves of P.L and red fruits of P.L data are 46 %, 67 %, 86%, 54%, 76%, 62%, 49% and 66 %, respectively. It is observed that the analyzed leaves of P.L is enriched in several essential elements such as K (8920 ± 18 mg/Kg), Ca (10465 ± 388 mg/Kg), Na (54.12 ± 4.92 mg/Kg) and Fe (203 ± 9 mg/Kg). Figures 3 and 4 illustrate the comparison study between the results obtained of two type Pistacia lentiscus L. plant red fruits and leaves.

Table 2: Quality assessment of the analytical results based on CRMs (NIST1573a and GSV4)

Element	NIST1573 a				GSV 4			
	This work	certified	U _{score}	Z _{score}	This work	certified	U _{score}	Z _{score}
As	ND	0.112±0.004	ND	ND	0.3±0.03	0.28±0.03	0.51	0.67
Ba	63.60±4.06	63	0.058	0.063	57.53±4.43	58±3	0.09	0.16
Br	1272±150	1300	0.117	0.147	3.39±0.26	3.4±0.4	0.03	0.04
Ca	ND	50500 ± 900	ND	ND	4128±239	4300±200	0.55	0.86
Ce	1.69 ±0.18	2	0.886	-1.033	1.03±0.08	1±0.1	0.27	0.35
Co	0.56 ±0.06	0.57 ± 0.02	0.158	-0.500	0.21±0.01	0.18±0.02	1.49	1.65
Cr	2.08 ±0.07	1.99 ± 0.06	0.976	1.500	1.01±0.14	0.8±0.02	1.44	10.25
Cs	0.005± 0.052	0.053	0.912	-6.000	0.29±0.01	0.29±0.02	0.17	0.20
Fe	360±17	368±7	0.444	0.140	240±16	0.03±0.001	1.28	2.44
Hf	0.146±0.014	0.14	0.396	0.476	ND	0.033	ND	ND
K	2.76 ±0.10	27000±500	1.177	1.200	17077±205	16600±600	0.75	0.80
La	2.51±0.16	2.3	0.552	0.609	0.59±0.02	0.6 ± 0.03	0.33	0.40
Na	114.85±10.46	136±4	1.889	5.288	ND	44±4	ND	ND
Rb	14.61±0.83	14.89±0.27	0.321	1.037	71.73±1.29	74±4	0.54	0.57
Sb	0.05±0.01	0.063±0.006	1.115	2.167	0.08±0.012	0.056±0.005	1.85	4.80
Sc	0.09±0.01	0.1	0.555	0.667	0.14±0.002	0.085±0.017	6.02	6.11
Sm	0.26±0.09	0.19	0.741	2.456	0.086±0.007	0.056±0.005	0.03	0.04
Sr	82.10±10.09	85	0.178	0.227	ND	15.2±0.5	ND	ND
Th	ND	ND	ND	ND	0.061±0.006	0.058±0.009	0.28	0.50
Zn	29.14±1.10	30.9±0.7	1.350	-2.514	26.10±0.705	26.3±0.9	0.17	0.22

Table 3: Results of concentration values obtained in (mg/kg) by k_0 -NAA technique

Element	leaves of P.L	red fruits of P.L	Ratio (red fruits/ leaves)	Element	leaves of P.L	red fruits of P.L	Ratio (red fruits/ leaves)
As	0.185±0.016	1.945±0.031	10	K	8920±18	17460±35	51
Ba	7.93±0.93	3.63±1	46	La	0.29±0.01	0.18±0.01	62
Br	10.49±0.04	7.07±0.05	67	Na	54.12±4.92	100±0.001	54
Ca	10465±388	3480±132	33	Rb	2.93±0.34	8.44±0.35	35
Ce	0.53±0.06	0.23±0.03	43	Sb	0.02±0.001	0.03±0.001	67
Co	0.089±0.01	0.10±0.007	86	Sc	0.095±0.002	0.063±0.001	66
Cr	1.6±0.05	2.27±0.14	70	Sm	0.041±0.004	0.02±0.001	49
Cs	0.066±0.009	0.061±0.007	93	Sr	31.13±3.80	6.49±1.26	21
Eu	0.01±0.001	ND		W	1.19±0.013	0.67±0.027	56
Mn	13.87±0.32	10.52±0.50	76	Th	0.069±0.008	ND	
Fe	203.2±9.14	84.12±5.55	41	Yb	0.02±0.001	ND	
Hf	0.03±0.001	ND		Zn	10.47±0.471	15.91±0.811	66

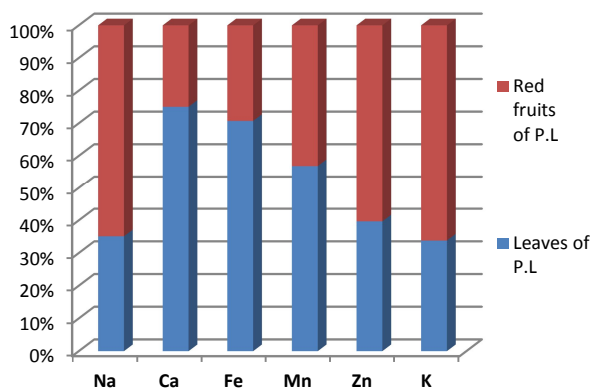


Fig. 3 Comparison study of analyzed essential elements of Pistacia lentiscus L. plant

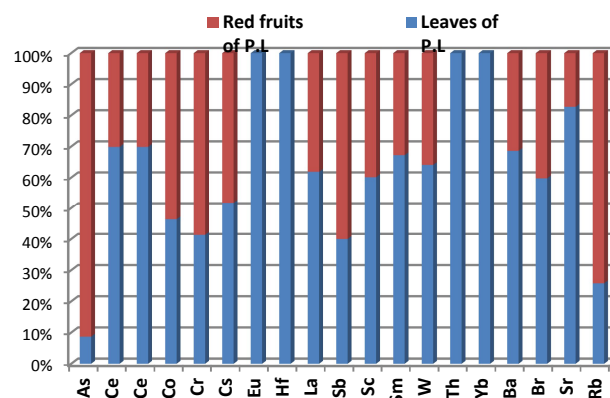


Fig. 4 Comparison study of analyzed non-essential elements of Pistacia lentiscus L. plant

VI. CONCLUSION

The data obtained on elemental concentration of the medicinal plants studied will be useful in deciding the dosage of the drugs prepared from these plant materials. The results of the present research work will be helpful to clinicians and scientists who would like to pursue further research in the areas of Pistacia lentiscus L. plant. The k_0 -Standardization neutron activation analysis method has proved to be a versatile tool to analyses biological materials like medicinal plants. The data obtained from this study can be used to evaluate the potentiality of these plants in their used for medicine. The analysis of these kinds of plant shows that the samples are adequate in some essential trace elements as well as for some of the nonessential trace elements. These trace elements are particularly significant as these foods are used in essential elements in nutrition, two major elements and twenty two minor and trace elements by k_0 -NAA techniques determined. In this study, irrelevant variations in the elemental composition of major elements with the exception of Na is less enriched 50 % in red fruits of P.L than leaves of P.L. Other elements are found relatively comparable except

the concentration values of Ba, Ca, Fe, and Sr are higher than in red fruits. The results of the present work will be used as a data base for the researchers and specialists. Due the high level of nutrients, recently more and more health care professionals are acknowledging the health benefits of many herbs and essential oils. Studies have shown vast and consistent results that bear up the traditional use of such natural medicines. Researchers are ever searching for the cures for what ails us, and many drugs have their origins in ethnic medicinal practices. In fact, a quarter of all pharmaceuticals contain botanicals.

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