



Evaluation of Analysis Results and Element Analysis of Salted Field Plant *Spergularia rubra* (L) J. Presl & C. Presl Using Chemometric Techniques

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Abstract. *Spergularia* (Caryophyllaceae) is seen as a cosmopolitan breed, ranging from temperate regions of all continents to sub-tropical climate regions, except for the Antarctic continent. Many of these strains have been investigated chemically and pharmacologically, such as hypoglycemic, diuretic, antihypertensive and cholesterol-lowering agents, antidiabetic, anti-cholinesterase antidiabetic agents. There are seven species of the genus second side being endemic in Turkey. In this study, all samples collected had been digested by microwave digestion system then element analysis of *Spergularia rubra* (L.) bitumen grown in the areas of Akdoğmuş, Çöl, Çaykaya, Gölgekonak and Üzümlük in Siirt and surrounding areas was carried out by ICP-OES. The highest values of Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Nb, Pb, Sb and Se were found to be 1.44, 1.62, 1.95, 8.38, 10.21, 5.78, 64.60, 1.51, 9.78, 2.87, 3.43 mg / L, respectively; Fe, K, Mg and Na values were found to be 1.56, 23.72, 14.82, 33.66 g / L. The highest concentrations of Na and K were found in Akdoğmuş and the concentrations of Fe and Mg were found in the Çöl. Chemometric techniques such as correlation, PCA and CA have been applied to saline areas and elemental contents. In the chemometric study, it was seen that the correlations at 99% level were very high. From the principle component analysis (PCA), the first two eigenvalues were found to be ≥ 1 . Two factors were found after maximal rotation and commonality.

Keywords: *Spergularia rubra*, ICP-OES, Saline plant.

Tuzlu Alan Bitkisi Olan *Spergularia rubra* (L) J. Presl & C. Presl'in Element Analizi ve Analiz Sonuçlarının Kemometrik Teknikler Kullanılarak Değerlendirilmesi

Özet. *Spergularia* (Caryophyllaceae), Antarktika kıtası dışında, tüm kıtaların ılıman bölgelerinden subtropikal iklim bölgelerine kadar dağılan kozmopolit bir cins olarak görülmektedir. Bu cinsin birçok türü Hipoglisemik, diüretik, antihipertansiyon ve kolesterol düşürücü, antidiyabetik, anti-kolinesteraz antidiyabetik gibi kimyasal ve farmakolojik açıdan incelenmiştir. Türkiye'de bu cinse ait 2 si endemik olmak üzere toplam 7 tür bulunmaktadır.

Bu çalışmada, Siirt ve çevresinde bulunan Akdoğmuş, Çöl, Çaykaya, Gölgekonak ve Üzümlük tuzlu alanlarında yetişen *Spergularia rubra* bitkisinin çözünürleştirme işlemleri mikro dalga yakma sistemi ile yapıldıktan sonra element analizi ICP-OES ile yapılmıştır. Bu bitkide bulunan Be, Cd, Co, Cr, Cu, Li, Mn, Mo, N, Pb, Sb ve Se en yüksek değerleri sırasıyla 1.44, 1.62, 1.95, 8.38, 10.21, 5.78, 64.60, 1.51, 9.78, 7.94, 2.87, 3.43 mg/L olarak bulunurken; Fe, K Mg ve Na değerleri 1.56, 23.72, 14.82, 33.66 g/ L olarak bulunmuştur. K ve Na konstanrasyonlarının en yüksek olduğu yer Akdoğmuş, Fe ve Mg konsantrasyonlarının ise en yüksek olduğu yer Çöl (Bayraktepe) olarak tespit edilmiştir. Korelasyon, PCA ve CA gibi kemometrik teknikler tuzlu alanlara ve element içeriklerine uygulanmıştır. Kemometrik çalışmada, % 99 seviyesindeki

korelasyonların çok yüksek olduğu görülmüştür. çoklu bileşen analizinden (PCA), ilk iki özdeğer ≥ 1 olarak bulunmuştur. Maksimal rotasyon ardından bulunan iki faktör değerlendirilmiştir.

Anahtar Kelimeler: *Spergularia rubra*, ICP-OES, Tuzlu alan bitki.

1. INTRODUCTION

Spergularia (Caryophyllaceae) is distributed from temperate regions of all continents to subtropical climatic regions except the Antarctic continent [1]. Many of these strains have been studied in terms of chemical and pharmacological aspects such as hypoglycemic [2], diuretic [3], antihypertensive and cholesterol-lowering agents [4], antidiabetic and anti-cholinesterase antidiabetic agents [5].

Spergularia rubra antenna fragments are widely used in the preparation of a consumed infusion for diuretic properties. However, the literature on this ongoing chemical compound is scarce and, despite the identification of certain compounds, quantitation data have not been reported. For example, in *S. rubra*, Zibareva et al. The phytoectoides reported by him have been discovered. In addition, some di-C-glycosyl-flavones have been isolated from this species and their structures have been elucidated by spectroscopic means. Such a compound exhibits a broad spectrum of activity, such as antibiotics, pancreatic lipase inhibitors, a radical scavenger, cholinesterase inhibition, and anti-hyperglycemic [6]. 6, 8-di-C- β -d-glucopyranosylapigenin, luteolin and chrysoeriol were obtained from the chemical isolation of the *Spergularia rubra* plant. Two new compounds were characterized based on their ¹H NMR and MS spectra and comparison with synthetic examples such as 7,2'-di-O- β -D-glucopyranosyl-1-6- α -l-arabinopyranosylpiperidine and 6- α -arabinopyranosylpiperine [7].

A new iced tea production involving a hydroethanolic extract of *Spergularia rubra* (L.) J. Presl & amp; C. has been studied to provide beverages, nutrients to consumers, special health-promoting functionality and desirable sensory properties. Types that contain both naturally occurring and added components. The phenolic

compounds of *S. rubra* hydroethanolic extract and iced tea were determined by HPLC-DAD. C-glycosyl flavones acylated with aliphatic acids, mainly represented by the apigenin derivatives, have been identified, 30 compounds containing unglycosylated C-glycosyl flavones containing C-glycosyl flavones acylated with acetic acid and C-glycosyl flavones acylated with aromatic acids. A strong inhibition of alpha-glucosidase, acetylcholinesterase and butyrylcholinesterase has been observed. The pH variation significantly decreased metabolite content and enzyme inhibitor capacity ($p < 0.05$). However, the drink enriched with *S. rubra* extract provides a valuable contribution to the health and nutrition of the consumer when there is less activity lost than that confirmed for basic iced tea. These results show that drinking this drink can be of potential interest for a variety of chronic disorders, especially Alzheimer's disease [8].

There are seven species of the genus *Spergularia* in Turkey. Two of these are endemic (*Spergularia lycia* and *Spergularia*) [9].

In this study, element analysis of *Spergularia rubra* (L.) bitumen grown in salty areas of Akdoğmuş, Çöl, Çaykaya, Gölgekonak and Üzümlük villages in and around Siirt was carried out by ICP-OES. Analyzes of Be, Cd, Co, Cr, Cu, Li, Mn, Mo, N, Pb, Sb and Se were done in this plant. In addition, chemometric techniques such as correlation with saline fields and elemental contents, multiple component analysis (PCA) and clustering analysis (CA) have been applied.

2. EXPERIMENTAL

2.1. Apparatus

Memmert brand mill was used in milling the collected plant samples. Berghof Speedwave MWS-3 micro-wave furnace was used for

solubilization of plant samples. The operating conditions of the microwave are given in Table 1.

Table 1. Microwave Operating Conditions.

Step	1	2	3	4
Temperature(°C)	150	190	100	100
Time (min)	10	10	10	10
Ta ^a (min)	5	10	10	5

The metal contents of the plant samples were made with Perkin Elmer ICP-OES Optima 700 DV. The operating conditions of ICP-OES are given in Table 2.

Table 2.. ICP-OES Operating Conditions.

View	Axial –Radial
Optical System	Echelle
Power	1450 W
Plasma Gas Flow	15 L min ⁻¹
Auxiliary Gas Flow	0.2 L min ⁻¹
Detector	Liquid State Detector
Sample Flow Rate	1.5 mL min ⁻¹
Nebulizer	Cyclonic
Nebulizer	Concentric Glass (Meinhard) Type A
Integration time	1.0 second

2.2. Reagents and solutions

The plant used in this study was collected in the salty areas of Akdoğmuş, Çöl, Çaykaya,

Gölgelikonak and Üzümlük villages in and around Siirt. The areas and locations of plant samples are given in Table 3.

Table 3. Location of samples taken.

Locations of Samples	Latitude N	Longitude E	Height (m)
Üzümlük	37°46' 69,90"	42°5' 44,3"	730
Gölgelikonak	37°46'21.46"	42° 6'53.96"	768
Akdoğmuş	37°51'38.27"	42° 3,06 '6.58"	622
Çöl (Bayraktepe)	37°51'30.58"	42° 4'56.52"	695
Çaykaya	37°04'39,07"	41° 45 '23,78"	598

The plant used in this study was dried in the shade at room temperature. Milled in a Memert brand mill. We weighed 0.6-1.0 g of plant samples and analyzed by microwave using HNO₃ / H₂O₂. All solutions were prepared from analytical grade Merck liquid and solid agents. Double distilled water was used as solvent water. Standard solutions used for ICP, 1000 ppm Merck brand standard solutions were used.

3. RESULTS AND DISCUSSIONS

3.1. Elemental analysis of plant samples

As shown in Table 1, plant samples taken from different regions of the saline fields were solubilized with microwave assistant. Solubilized samples Completed with a certain volume of pure water and analyzed by ICP-OES. The results of the analysis are given in Table 4.

Table 4. Results of analysis of plant samples taken from different regions of the plots by ICP-OES (mg/kg).

Locations	Be	Cd	Co	Cr	Cu	Fe	K	Li
Akdoğan	1.44	1.50	1.56	5.25	8.24	999.73	23716.32	4.87
Çöl	1.19	1.62	1.95	8.38	10.21	1564.30	9845.10	5.78
Çaykaya	n.d.	n.d.	0.03	0.29	0.28	46.84	258.30	0.15
Gölgelikonak	n.d.	n.d.	0.09	0.59	0.67	119.70	352.60	0.54
Üzümlük	n.d.	n.d.	0.04	0.33	0.40	83.06	222.20	0.28

Locations	Mg	Mn	Mo	Na	Ni	Pb	Sb	Se
Akdoğan	9483.72	53.02	1.44	33657.45	6.18	2.75	2.87	3.43
Çöl	14824.39	64.57	1.51	20344.02	9.78	7.94	2.49	2.59
Çaykaya	162.40	3.49	0.01	1083.00	0.41	0.05	0.02	n.d.
Gölgelikonak	271.20	3.72	n.d.	871.60	1.03	0.43	0.01	n.d.
Üzümlük	200.30	3.44	n.d.	812.80	0.56	0.10	0.01	n.d.

The levels of Be, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na and Ni metals in plant samples are compared with the values recommended by the Turkish standard [10] (200 mg L⁻¹ for Ca, 12 mg L⁻¹ for K, 175 mg L⁻¹ for Na and 50 mg L⁻¹ for Mg). It is seen that all Ca, K, Na and Mg levels belonging to Gölgelikonak, Üzümlük, Akdoğan, Desert and Çaykaya results are higher than the values suggested according to Turkish standards [10].

3.2. Correlation Analysis

Correlation coefficients between elements analyzed in plant samples are calculated as the correlation matrix and given in Table 5. Element correlation data were analyzed in terms of linear correlation coefficients which were significant in the 95% confidence interval and 99% confidence interval.

Table 5. Correlation matrix for element concentrations in plant samples.

	Be	Cd	Co	Cr	Cu	Fe	K	Li	Mg	Mn	Mo	Na	Ni	Pb	Sb	Se
Be	1.000															
Cd	0.985**	1.000														
Co	0.965**	0.995**	1.000													
Cr	0.910*	0.967**	0.987**	1.000												
Cu	0.965**	0.995**	1.000**	0.987**	1.000											
Fe	0.914*	0.970**	0.989**	1.000**	0.989**	1.000										
K	0.930*	0.854	0.801	0.695	0.801	0.702	1.000									
Li	0.971**	0.996**	0.999**	0.982**	0.999**	0.984**	0.816	1.000								
Mg	0.920*	0.974**	0.991**	0.999**	0.990**	0.999**	0.712	0.986**	1.000							
Mn	0.968**	0.997**	1.000**	0.985**	1.000**	0.986**	0.807	0.999**	0.989**	1.000						
Mo	0.988**	1.000**	0.994**	0.963**	0.993**	0.965**	0.862	0.995**	0.970**	0.995**	1.000					
Na	0.981**	0.933*	0.894*	0.811	0.895*	0.817	0.984**	0.905*	0.826	0.899*	0.939*	1.000				
Ni	0.908*	0.966**	0.986**	1.000**	0.986**	1.000**	0.693	0.982**	0.998**	0.984**	0.961**	0.809	1.000			
Pb	0.765	0.864	0.908*	0.963**	0.908*	0.960**	0.476	0.896*	0.956*	0.903*	0.855	0.625	0.964**	1.000		
Sb	1.000**	0.990**	0.971**	0.921*	0.972**	0.925*	0.920*	0.977**	0.931*	0.974**	0.992**	0.975**	0.919*	0.783	1.000	
Se	0.998**	0.974**	0.948*	0.885*	0.948*	0.889*	0.950*	0.956*	0.896*	0.952*	0.978**	0.990**	0.883*	0.727	0.996**	1.000

** . Correlation is significant at the 0.01 level (2-tailed).

* . Correlation is significant at the 0.05 level (2-tailed).

As shown in Table 5, we can summarize the correlation coefficients between the elements as follows:

- Cd with Be (0.985)
- Co with Be (0.965) and Cd (0.995)
- Cr with Cd (0.967) and Co (0.987)
- Cu with Be (0.965), Cd (0.995), Co (1.000) and Cr (0.987)
- Fe with Cd (0.970), Co (0.989), Cr (1.000) and, Cu (0.989)
- Li with Be (0.971), Cd (0.996), Co (0.999), Cr (0.982) and Cu (0.999)
- Mg with Cd (0.974), Co (0.991), Cr (0.999), Cu (0.990) and Li (0.986)
- Mn with Be (0.968), Cd (0.997), Co (1.000), Cr (0.985), Cu (1.000), Li (0.999) and Mg (0.989)
- Mo with Be (0.988), Cd (1.000), Co (0.994), Cr (0.963), Cu (0.993), Li (0.995), Mg (0.970) and Mn (0.995)
- Na with Be (0.981), K (0.984)
- Ni with Cd (0.966), Cu (0.986), Fe (1.000), Li (0.982), Mg (0.988), Fe (1.000), Mn (0.995) and Mo (0.961)
- Pb with Cr (0.963), Fe (0.963) and Ni (0.964)
- Sb with Be (1.000), Cd (0.990), Co (0.971), Cu (0.972), Li (0.977), Mg (0.974), Mn (0.992) and Na (0.975)

As we have seen above, there is a very high and significant correlation at the 99% confidence level.

3.3. Principal component analysis (PCA)

Stronger chemometric techniques such as principal component analysis (PCA) have also been studied to further explain the locations of plant specimens and the elements the plant contains.

The principal component analysis (PCA) was performed on the correlation matrix using the IBM SPSS statistic V22 package (Release 22.0.0.0, 1989-2003) and Minitab 17 (Licensing: 17.1.0.0, 2013). The principal component analysis was applied to the data matrix of plant samples and the concentrations of the contained elements (16 x 5 states). The results show that two eigenvalues > 1 account for 99.98% of the total variance, and the second eigenvalue accounts for about 45.27% of the variance. The main component load for plant samples yields two components, which describe approximately 99.98% of the total variance with 54.71% and 45.27% of the contribution of each factor, respectively. For this reason, the first two eigenvalues were selected for further analysis. The maximum two-factor load after the rotation and communalities are given in Table 6. Factor 1 has high loads for Pb, Ni, Cr, Fe, Mg, Co, Cu, Mn, Li, Cd and Mo, which accounts for about 54.71 of the total variance. Factor 2 is loaded with K, Na, Se, Be, Sb and accounts for 45.27% of the total variance. A 2D drawing of the PCA loads is shown in Figure 1 and the relationships between the elements are readily apparent.

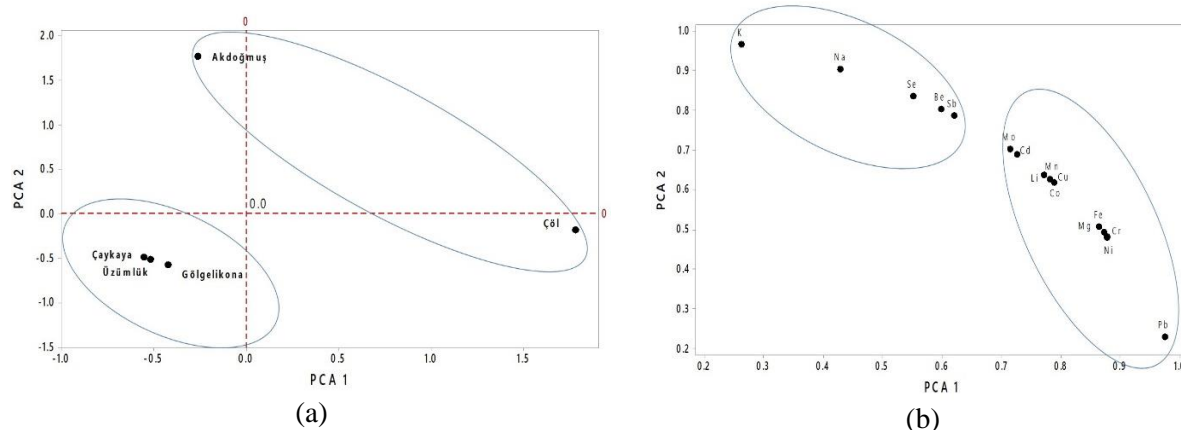


Figure 1. Score ve loading plots (a score plots ve b loading plots).

Table 6. The first two rotated main component loadings and scores.

Element	The loading		Vocational	The score	
	PC1	PC2		PC1	PC2
Pb	0.973	0.230	Akdoğmuş	-0.267	1.768
Ni	0.877	0.480	Çöl	1.777	-0.180
Cr	0.876	0.483	Çaykaya	-0.561	-0.494
Fe	0.871	0.491	Gölgelikonak	-0.426	-0.573
Mg	0.863	0.505	Üzümlük	-0.523	-0.520
Co	0.787	0.617			
Cu	0.787	0.617			
Mn	0.780	0.626			
Li	0.770	0.637			
Cd	0.725	0.689			
Mo	0.713	0.701			
K	0.261	0.965			
Na	0.428	0.904			
Se	0.550	0.835			
Be	0.597	0.802			
Sb	0.619	0.786			
Eigenvalue	8.75	7.24			
Variance (%)	54.71	45.27			
Cumulative (%)	54.71	99.98			

3.4. Cluster (CA) Analysis

This technique is a classification process that includes a measure of the similarity between the objects to be crowned [11]. Plant samples are grouped into clusters in terms of similarity.

The measure of the similarity is based on the Euclidean distance between the values. The clustering method used is Ward's method. Clustering analysis was performed using the Minitab 17 (Licensing: 17.1.0.0, 2013) Statistics package.

The results obtained in the clustering analysis are given in Figure 2-3. These clusters represent the similarity between the origins of metals for plant specimens. Similar results were obtained with Principle component analysis results.

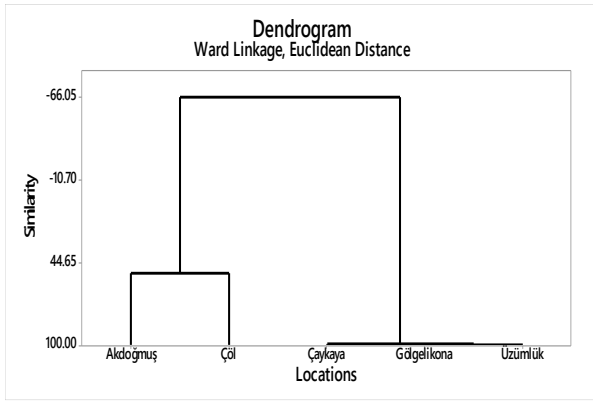


Figure 2. Clustering dendrogram of collected places of plant specimens.

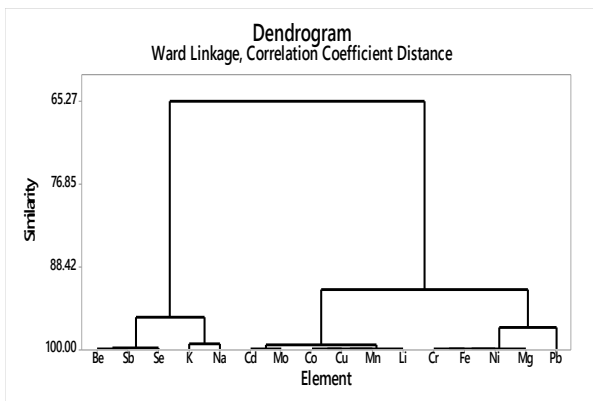


Figure 3. Clustering dendrogram of element concentrations.

4. CONCLUSION

In this study elemental analysis of *Spergularia rubra* (L.) plant, taken from the saline areas of Akdoğan, Çöl, Çaykaya, Gölgekonak and Üzümlük, was carried out.

The highest concentrations of Na and K were found in Akdoğan and the concentrations of Fe and Mg were found in the Çöl. In the chemometric study, it was seen that the correlations at 99% level were very high. From the principle component analysis (PCA), the first two eigenvalues were found to be ≥ 1 . Two factors were found after maximal rotation and commonality.

In Factor 1, Pb, Ni, Cr, Fe, Mg, Co, Cu, Mn, Li, Cd and Mo were found, which accounted for about 54.71 of the total variance. In Factor 2, K, Na, Se, Be and Sb are found and explain 45.27% of the total variance.

Clustering (CA) analysis was also found to be parallel to PCA analysis. It is seen that Akdoğan and Çöl showed similar characteristics from the places where the plant samples were taken, and Çaykaya, Gölgekonak and Üzümlük are similar. When analyzing CA analysis for element analysis, it will be seen that two groups are separated as seen in PCA analysis.

In the first group, Be, Sb, Se, K and Na are included while in the second group, Cd, Mo, Co, Cu, Mn, Li, Cr, Fe, Ni, Mg and Pb are included.

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