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EVALUATION OF CHEMICAL CONTENT AND RADICAL SCAVENGING ACTIVITY OF *ALLIUM VINEALE* L. EXTRACT AND ITS ELEMENTAL ANALYSIS

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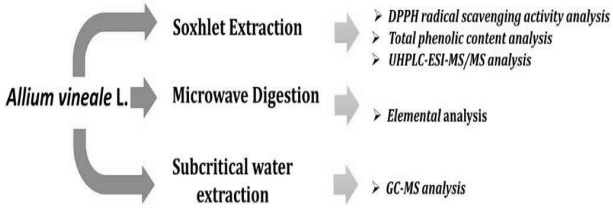
Allium vineale L. was extensively analysed to determine its radical scavenging activity, chemical composition, total polyphenol content, and elemental composition. The Soxhlet and the subcritical water extraction methods were performed for the extraction procedure. The extracts obtained in the subcritical water extraction method were evaluated by GC-MS. 11 compounds were determined according to mass spectral libraries of GC-MS, Wiley7Nist05.L, NIST05a.L, W9N11.L. The obtained compounds were evaluated for their benefits on health according to literature. DPPH inhibition of *Allium vineale* L. was obtained as 58.60 % and the total polyphenol content of *Allium vineale* L. was obtained as 12 µg/mL gallic acid equivalent. 28 elements were obtained in the elemental analysis by ICP-MS, using the microwave digestion procedure. UHPLC-ESI-MS/MS analysis was performed for the evaluation of methanolic extract of *Allium vineale* L. obtained in the Soxhlet extraction method.



INTRODUCTION

Allium species (*Liliaceous*) have been widely used for alternative medicine almost all over the world since ancient times.[1–5] Investigations have shown that *Allium* species are beneficial and may be effective in preventing cardiovascular diseases, tumour promotion, and ageing.[2–5] Several *Allium* species of both cultivated species and wild species from various locations have been evaluated to enlighten their antioxidative potentials by researchers.[6] Besides, antioxidative properties[7,8] of leaves of various wild and grown *Allium* sorts were examined for their activities of antioxidant enzymes (superoxide dismutase, catalase, peroxidase, and glutathione peroxidase), hydroxyl radicals and reduced glutathione, and quantities of malonyldialdehyde superoxide.[9]

The antibacterial activity of *Allium vineale* L. (*A. vineale*) was studied and was found to be at a quite high level.[10] In addition, organic solvent extracts (methanolic and ethanolic extracts, etc.) of this plant can be used as natural antibacterial additives for the incorporation in cheese and various food products.[10]

Allium genus is a natural food and consumed by many communities. There are 198 natural taxa belonging to the genus *Allium* in Turkey. *A. vineale* grows in Siirt, Van and Hakkari provinces in Turkey and it is regionally called Sırık or Sırma. *A. vineale* have been used for herbed cheese production in Turkey for many centuries.[10] It is consumed by citizens because it gives a nice taste and a pleasant smell when mixed with cheese.[11,12]

In the present study, the subcritical water extraction (SWE) method was performed to

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determine the chemical content of *A. vineale*. SWE is carried out under the temperature conditions between 100-374 °C and under sufficient pressure conditions to keep the water in the liquid phase.[13] SWE method has advantages of being environmentally friendly, very cheap, easy to find, not toxic and it does not leave organic waste.[13] Total phenolic contents of *A. vineale* and DPPH radical scavenging activity were spectrophotometrically determined. The presence of several phenolic acids in the *A. vineale* was determined using UHPLC analysis. Phenolic contents and fatty acid compositions were determined by LC-MS/MS and GC-MS.[14–16] ICP-MS, which is a type of mass spectroscopy and is highly sensitive and allows the determination of metals and several non-metals at a concentration level of ppb, was used for elemental determinations.[17]

MATERIAL AND METHOD

Chemicals and Instruments

The Soxhlet apparatus was used to extract *A. vineale* and the rotary evaporator (Hei-VAP, Heidolph Instruments, Germany) was used to concentrate the extract samples. Phenolic content compositions were determined by LC-MS/MS (Shimadzu, Kyoto, Japan).[16] The Berghof Speedwawe MWS-3 model microwave was used in the digestion process of *A. vineale*. The Thermo Scientific brand I CAP Q model ICP-MS instrument was used for the metal analysis of the plant. HNO_3 and H_2O_2, 2,2-diphenyl-1-picrylhydrazyl (DPPH•), Folin-Ciocalteau's phenol reagent, gallic acid, C_7-C_{40} saturated alkane standard mixture and ascorbic acid were obtained from Sigma Sigma-Aldrich (St. Louis, MO). All other reagents were of analytical grade. The UV mini-1240 spectrophotometer (Shimadzu, Duisburg, Germany) was used to measure the total phenolic content and the radical scavenging activity (DPPH). SWE experiments were done using a stainless steel cylindrical extractor (100 mm × 5 mm i.d.). Both ends of the extractor were covered with 0.45 μm mesh size filters to prevent the particulates. The experimental set-up as shown in previous work was used for SWE extraction experiments.[18] The Soxhlet apparatus was used in the Soxhlet extraction process.

Plant material

The plant of *A. vineale* was collected in Eruh district of Siirt of south eastern Anatolia region of Turkey province in April 2015 and identified by Dr Mehmet Fidan. This specimen has been stored at the Herbarium of Siirt University (Herbarium plant number: MeF 2427). The collected plant samples were dried, powdered and then stored in a jar for further analysis.

The procedure of the subcritical water extraction method

In the other part of this study, samples to be extracted were prepared by adding the mixture of 0.5 g dried and powdered *A. Vineale* sample and 0.5 g of sea sand into the extraction cell. The extraction cell was covered by glass wool, screwed on, placed into the oven and pressurized with water. After static mode extraction (30 min), the exit valve was opened and the extracts were collected in ultra-pure water during dynamic mode extraction. 5 mL of water was used in SWE for each experiment. SWE experiments were performed at 105 °C under 40 bar of N_2 gas in the constant pressure mode. The solvent of the obtained extract was evaporated by the rotary evaporator and residue was dissolved in methanol.

GC-MS Analysis

The methanolic extract of *A. vineale* was analysed by GC-MS to identify its chemical composition. GC-MS was operated by the method given in the previous work[18]. A certified reference material, C_7–C_{40} saturated alkane mixture, which contains each C_7–C_{40} component in a concentration of 1000 μg in mL of hexane was used to determine the Kováts index of identified compounds.

The procedure of the Soxhlet extraction method

Samples of *A. vineale* were subjected to the Soxhlet extraction method for analysis of phenolic compounds by LC-MS/MS. 10 g of dried and powdered plant sample was subjected to a Soxhlet extraction system containing 160 mL of 80:20 methanol-water mixture as a solvent. LC-MS/MS was operated using the method given by Ertaş *et al.* to determine phenolic content composition.[16]

Extract Preparation of A. vineale for total phenolic compounds and DPPH radical scavenging activity analysis

A mixture of 10 g of dried and powdered *A. vineale* sample and 100 mL of the methanol-

water mixture (80:20%) was shaken at 37 ^{o}C for 24 hours. Next, the mixture was filtered through rough filter paper into a glass container. Then, Methanol was removed by a rotary evaporator and the amount of residue was determined. After that, the concentration of each plant sample was dissolved again with methanol to adjust the concentration to be 10 mg/mL. Finally, the samples were kept in the refrigerator for further analyses.

Total phenolic content analysis

The total phenolic contents of plant materials were determined using the Folin-Ciocalteau's as a reagent and gallic acid as standard. This method relies on the reaction of phenolic contents with the Folin-Ciocalteau's reagent and phenolic contents were determined by measuring the blue colour of the formed compound in the visible section of the spectrum [19]. An aliquot (0.2 mL) of extracts and Folin-Ciocalteau's reagent (1 mL) were added to a volumetric flask. After incubation for 6 min at room temperature, 1 mL of 7% Na_2CO_3 solution was added and the mixture was shaken. The absorbance of the mixed solution was measured at 760 nm with a UV mini-1240 Spectrophotometer. The same procedure was repeated with gallic acid solutions (8-200 mg/mL). All samples were analysed in 3 replicates. The amount of the total phenolic content was calculated using a standard curve (R^2 = 0.9994) of gallic acid constituted by using the concentration of each gallic acid solution (0–0.2 mg/mL) against its absorbance.

DPPH analysis

The DPPH assay was performed according to the method explained by Villaño *et al.*[20] Briefly, a solution of 0.5 mL of sample and 2 mL of 0.01 mM DPPH (dissolved in methanol) were stirred and incubated for 30 min in dark. The absorbance of the mixture was measured at 517 nm with a UV mini-1240 Spectrophotometer. The percentage inhibition was calculated by the formula given below.

$$\text{Inhibition \%} = \left(\frac{A_c - A_i}{A_c} x100 \right)$$

where A_c is the control absorbance and A_i is the absorbance values of the sample.

Microwave Digestion Process of A. vineale

The digestion process of the plant sample was performed by the Berghof Speedwawe MWS-3 model microwave digestion apparatus under the operating conditions given in Table 1. In this process, 0.5 g part of the dried plant was weighed and put into the pressure-resistant PTFE (Polytetrafluoroethylene) vessel. 10:2 mL of HNO_3: H_2O_2 acid mixture was added to the plant sample. The digestion process was operated under conditions given in Table 1.

RESULTS AND DISCUSSION

The chemical composition of A. vineale extracts obtained by SWE

SWE of *A. vineale* plant was performed under optimum conditions. Extracts were collected after a specific time under 105 °C and 40 bar. To determine extracted compounds, samples were analysed by GC-MS according to Wiley7Nist05.L, NIST05a.L, W9N11.L. The determined compounds are given in Table 2. Though characteristics of these compounds vary on a wide range, they may show antioxidative activity one by one or collectively. Some of the compounds given in Table 2 were evaluated below according to literature. Krist *et al.* enlightened the volatile compounds of a series poppy seed oil. Ethanoic acid, which was found in our study (component number of 1 in Table 2), is one of the obtained compounds in their work with retention index of 660.[21] Also, Pino *et al.* identified ethanoic acid as a component of *Morinda citrifolia* L.[22] Aly *et al.* determined 3-amino-2-ethylbutanoic acid study (component number of 6 in Table 2) in the GC-MS analysis of ethyl acetate fraction extracted from *Dalbergia Sissoo*.[23] According to the work done by Mochizuki *et al.*, in which *Allium sativum* L. was comprehensively analysed, it was found that *Allium sativum* L. has antibacterial activity, antifungal activity, virucidal activity, etc. due to containing active sulfur compounds.[24] Herein, we determined dimethyl trisulfide (component number of 7 in Table 2) as a sulphurous compound of *A. vineale*. 2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one (component number of 8 in Table 2) was found as one of the components of ether extract of *Panax ginseng*, which was proposed for

its antifungal and antitumor properties by Xu *et al.*[25] p-vinylguaiacol (2-methoxy-4-vinylphenol) (component number of 9 in Table 2) was detected by Asuming *et al.* In the investigation of the essential oil composition of four *Lomatium* Raf. Species.[26] The retention index, found for this compound by Asuming *et al.*, is quite close to our findings.

Total phenolic content analysis

The total phenolic amount was found as a result of the method applied to the extract obtained from *A. vineale*, as indicated in literature,[12] and the obtained values are given in Table 3. Besides, the amount of antioxidant was analysed as given in literature[13] and the obtained result is given in Table 3.

Quantitative analysis of phenolic compounds

As indicated in literature[27,28] the UHPLC-ESI-MS/MS technique is used in the quantitative analysis of phenolic compounds. An accurate quantitative method was developed on a mass spectrometer equipped with a triple quadrupole analyser for the analyses of twenty-seven compounds.[14–16] The methanol extracts of *A. vineale* were analysed to quantify these compounds by the mentioned method. The obtained results are given in Table 4.

Table 1

Operating conditions of the digestion process

	1	**2**
T (^oC)	150	190
P bar	50	50
Ta (min) [a]	70	90
Time (min) [b]	5	5

[a] Waiting time at the desired temperature; [b] Time between the two sequential temperatures.

Table 2

GC-MS results of extracts obtained from SWE of *A. vineale*

C. No	**RT (min)**	**Peak Area (%)**	**Compound Name**	**Chemical Formula**	**Molecular weight (g/mol)**	**Peak Quality (%)**	**KI**	**RIL**	**Ref No.**
1	2.92	10.27	Ethanoic acid	$C_2H_4O_2$	60.05	86	nc	660	19
2	3.26	3.14	Glycidyl alcohol	$C_3H_6O_2$	74.08	58	nc	nd	nd
3	3.56	1.75	Glyoxalic acid	$C_2H_2O_3$	74.04	59	nc	nd	nd
4	4.64	1.41	Cyclobutanol	C_4H_8O	72.11	53	nc	nd	nd
5	5.96	1.41	Cyclopropyl carbinol	C_4H_8O	72.11	51	nc	nd	nd
6	7.59	4.32	3-amino-2-ethylbutanoic acid	$C_6H_{13}NO_2$	131.09	60	nc	nd	nd
7	8.46	3.10	Dimethyl trisulfide	$C_2H_6S_3$	126.26	91	975.64	972	20
8	14.19	9.27	2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one	$C_6H_8O_4$	144.13	68	1160.48	1162	21
9	21.93	9.67	p-Vinylguaiacol	$C_9H_{10}O_2$	150.07	94	1318.65	1315	22
10	22.74	1.54	Myocol	$C_{10}H_{13}N_5O_4$	267.24	53	1353.66	nd	nd
11	69.94	0.74	Gibberellic acid	$C_{19}H_{22}O_6$	346.37	55	3263.07	nd	nd

RT: Retention times, C. No: Component number, KI: Kovats index, RIL: Retention Index obtained from literature, nc: not calculated, nd: not detected

Table 3

The total phenolic content and DPPH radical scavenging activity of *A. vineale*

Sample (Locality)	**%DPPH Inhibi tion**	**Gallic acid equivalent µg/mL (Total phenolic)**
A. vineale (Siirt-Eruh/Turkey)	58.60	12.00

Table 4

Compounds of the methanolic extract of *A. vineale*

C. No	Compound	(µg analyte/kg extract)	SD %
1	Hesperidin	nd	nd
2	Coumarin	nd	nd
3	Quinic acid	14796.17	710.22
4	Malic acid	29904.61	1435.42
5	tr-Aconitic acid	11.4	0.55
6	Gallic acid	nd	nd
7	Chlorogenic acid	5.01	0.24
8	Protocatechuic acid	7.03	0.34
9	Tannic acid	219.76	10.55
10	tr-caffeic acid	5.19	0.25
11	Vanillin	4.32	0.21
12	Rosmarinic acid	nd	nd
13	p-Coumaric acid	179.56	8.62
14	Rutin	0.81	0.04
15	Hyperoside	213.83	10.26
16	Myricetin	nd	nd
17	Fisetin	nd	nd
18	4-OH Benzoic acid	nd	nd
19	Salicylic acid	nd	nd.
20	Quercetin	9.62	0.46
21	Kaempferol	41.79	2.01
22	Naringenin	nd	nd
23	Hesperetin	nd	nd
24	Luteolin	24.44	1.17
25	Apigenin	61.03	2.93
26	Rhamnetin	249.85	11.99
27	Chysin	nd	nd

nd: not detected.

Table 5

Elemental analyses of *A. vineale*

Li (ppb)	**Be** (ppb)	**Co** (ppb)	**As** (ppb)	**Se** (ppb)	**Cd** (ppb)	**Sb** (ppb)
242.85 ± 2.215	3.42 ± 0.27	132.48 ± 1.23	26.37 ± 1.32	93.28 ± 8.31	5.76 ± 2.49	4.24 ± 0.02
La (ppb)	**Ce** (ppb	**Tl** (ppb)	**Pb** (ppb)	**V** (ppb)	**Zn** (ppm	**Mn** (ppm
62.82 ± 0.23	85.18 ± 1.21	2.69 ± 0.11	180.32 ± 0.71	191.35 ± 3.57	7.75 ± 0.04	10.46 ± 0.12
Sr (ppm)	**Mo** (ppm)	**Fe** (ppm)	**Sn** (ppm)	**Cu** (ppm)	**Ba** (ppm)	**Na** (ppm)
6.51 ± 0.10	0.80 ± 0.03	75.50 ± 1.41	3.53 ± 0.31	4.27 ± 0.08	4.84 ± 0.07	291.20 ± 2.38
B (ppm)	**Mg** (ppm)	**P** (ppm)	**K** (ppm)	**Ti** (ppm)	**Ca** (ppm)	**Cr** (ppm)
10.99 ± 0.08	745.80 ± 13.33	1020.24 ± 11.30	1425.31 ± 4.95	3.78 ± 0.20	2046.87 ± 8.98	0.94 ± 0.03

The chromatogram of standard mix and chromatogram of *A. Vineale* methanol extracts as shown in Fig. 1 and Table 4, quinic and malic acids were found to be the most abundant compounds, (14.79 and 29.90 mg/g extract, respectively) in the methanol extracts of *A. vineale*. Furthermore, tr-aconitic acid, chlorogenic acid, protocatechuic acid, tannic acid, tr-caffeic acid, vanillin, p-coumaric acid, rutin, hyperoside, quercetin, kaempferol, luteolin, apigenin, and rhamnetin were also detected in the methanol extract of this plant (Table 4). Additionally, hesperidin, coumarin, gallic acid, rosmarinic acid, myricetin, fisetin, 4-OH benzoic acid, salicylic acid, quercetin, naringenin, hesperetin, and chysin were not identified in the methanol extract of *A. vineale*.

Elemental analysis of A. vineale

The elemental analysis of *A. Vineale* plant was performed using ICP-MS and the obtained results were given in Table 5.

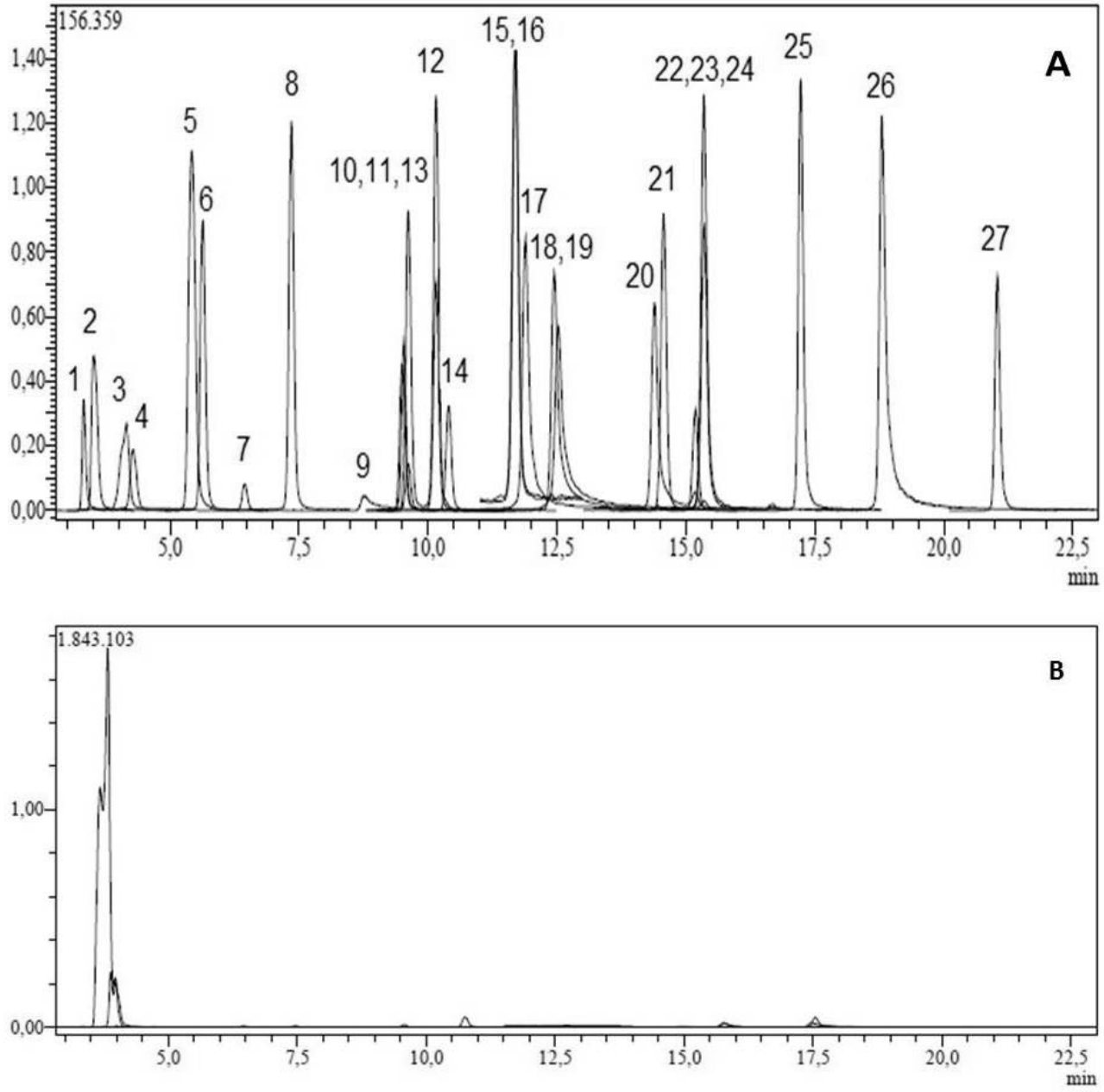

Fig. 1. A – UHPLC-ESI-MS/MS chromatogram of standard mix (1: quinic acid, 2: malic acid, 3: tr-aconitic acid, 4: gallic acid, 5: chlorogenic acid, 6: protocatechuic acid, 7: tannic acid, 8: tr- caffeicacid, 9: vanillin, 10: p-coumaric acid, 11: rosmarinic acid, 12: rutin, 13: hesperidin, 14: hyperoside, 15: 4-OH benzoic acid, 16: salicylic acid, 17: myricetin, 18: fisetin, 19: coumarin, 20: quercetin, 21: naringenin, 22: hesperetin, 23: luteolin, 24: kaempferol, 25: apigenin, 26: rhamnetin, 27: chrysin). **B**: UHPLC-ESI-MS/MS chromatogram of Allium Vineale L. methanol extract.

When Table 5 is examined, it is seen that the values of P, Fe, Na, Mg, and K are very high in mg/g level. While Ti, Cr, Mn, Cu; Zn, Sr, Mo, Sn, and Ba element values were found to be in the range of 1-10 ppm, element values of Li, Be, V, Se, Cd, Sb, La, Ce, Tl and Pb were determined at ppb level. While Cr and Fe values are parallel to the metal values in different vegetable and spice plants in literature[29–31], Co, Mn, Zn, Cu and Sr values are found to be lower.[30]

CONCLUSIONS

A. vineale plant, presented in this study, was collected from the rural district of Eruh in Siirt. In this study, the components of *A. vineale* were determined using the subcritical extraction method. Besides, the total phenolic amount, antioxidant capacity, phenolic compounds and metal values of the plant were studied. According to this study, quinic and malic acids (14.79 and 29.90 mg/g

extract, respectively) were the most abundant phenolic compounds in *A. vineale* extracts. The total phenolic content and the DPPH radical scavenging activity of *A. vineale* were found as 58.60 % and 12.00 µg/mL, respectively. The values of P, Fe, Na, K, and Mg were found to be the highest in the elemental analysis of Allium *A. vineale* using ICP-MS. However, Li, Be, V, Co, Ni, As, Se, Sb, La, Ce and Pb values were found to be very low at the ppb level.

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