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Synchrotron Radiation X-Ray Fluorescence Analysis in Studying Siberian Shrubs of an Urban Ecosystem

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Abstract—Synchrotron radiation X-ray fluorescence analysis (SR XRFA) is used to study the elemental composition of three shrubs of two genera, *Spiraea* and *Myricaria*, growing in the urban ecosystem of Novosibirsk and under background conditions. At least 20 elements are identified. The most severe pollution with heavy metal under urban conditions is observed in plants of *Myricaria bracteata*. *Spiraea chamaedryfolia*, which is more tolerant to pollution under urban conditions, can be recommended for use as a standard, while *M. bracteata* is recommended as a bioindicator plant for environmental pollution. The data obtained for the elemental composition of plant and soil specimens can be added to databases.

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INTRODUCTION

X-ray fluorescence (XRFA) spectroscopy is a proven analytical way of assessing chemical elements qualitatively and quantitatively. It is a multielement, simultaneous, nondestructive technique that is entirely suited for analyzing parts of the environment, including plants [1]. The use of synchrotron radiation (SR) substantially improves the capabilities of the technique, since the brightness of SR sources allows us to shorten the time of acquiring an experimental spectrum considerably, improving its rapidity and sensitivity. The possibility of rearranging the energy of excitation in the range of a station's working energies [2] opens up additional prospects when analyzing the concentrations of chemical elements that can act as markers for different species of plants and pollutants. In combination with the relative simplicity of processing the experimental spectra, the possibility of simultaneously determining many elements with high sensitivity and good accuracy [3, 4] allow us to quickly obtain data on the composition of specimens. However, the fairly rare use of XRFA for analyzing plant material is mainly due to the insufficient number of certified samples that can act as external reference standards [5]. Expanding the database on the chemical composition of plants and searching for optimum reference samples (standards) could therefore help solve the problem of using SR XRFA in botanical and environmental research. SR XRFA also requires only small weighed amounts of a material for analysis, which is especially relevant when studying the chemical elemental composition of plant and soil specimens. It also allows us to determine the entire set of chemical elements in one specimen of a material, thus offering the possibility of making an adequate comparative analysis. We can also determine the concentrations of elements over a wide range with no chemical preparation of specimens, which eliminates errors created by adding or removing elements along with chemicals.

Analyzing microelements is a fundamental problem in the environmental sciences. The quality and safety of ecosystems and the levels of anthropogenic pollution are determined from the concentrations of microelements that exceed the maximum allowable concentrations (MACs) in the environment [6]. Many authors use ligneous plants or parts of them as bioindicators for studying urbanized environments [7]. Plants demonstrate different resistances to heavy metal pollution, depending on the species. It is therefore important to search for species of plants that are both tolerant to an excess of pollutants and sensitive to technogenic stress, in order to determine the effect the anthropogenic factor has on plants.

Species of the genera *Spiraea* L. and *Myricaria* Desv. are environmentally flexible, resistant to gases, decorative, grow well, blossom under the conditions of urban environment, and are widely used in green construction [8]. Their properties as bioindicators for the pollution of the environment were noted in [9]. From our viewpoint, literature data on the concentrations of chemical elements of plants from the genera *Spiraea* and *Myricaria* are sparse and do not offer fair representation of different taxons.

The aim of this work was to determine the compositions and concentrations of elements of shrubs from the genera *Spiraea* and *Myricaria* under background conditions and in the urban ecosystem of Novosibirsk via SR XRFA, in order to identify the species the most resistant to anthropogenic pollution and determine the possibility of using plant specimens as standards.

EXPERIMENTAL

Specimens of two species of spirea—*Spiraea chamaedryfolia* L. (germander meadowsweet) and *S. media* Schmidt (Russian spirea) from the family Rosaceae—plus *Myricaria bracteata* Royle from the family Tamaricaceae were our objects of study. Plants from Memorial of Glory Square in the Lenin district of Novosibirsk, one of the city's most environmentally unfriendly areas (urban conditions), were chosen for the study. The Lenin district is one of the worst in terms of atmospheric emissions of carbon oxide, sulfur dioxide, nitrogen oxides, and solid pollutants. The general level of atmospheric pollution in Novosibirsk is classified as "Elevated." [10]. Individual plants of the same age growing in a forested area within the territory of the Russian Academy of Sciences' Central Siberian Botanical Garden, located in the relatively environmentally friendly Soviet district (Akademgorodok) were taken as our control plants.

Our plant specimens were selected on June 27, 2019, during the reproductive season. They were gathered uniformly around their crowns and simultaneously in both areas. Ten annual twigs were collected from each plant, and average specimens were created. Leaves and stems from the plants and samples of soils from their habitats were analyzed. An average specimen was composed of five to ten individual plants [11]. Envelope specimens of soil were taken from each root's habitable layer (10–15 cm).

Weighed amounts of air-dried plant feedstock and soils (1 g) were ground in an agate mortar. Samples were then pressed in the form of tablets ~1 cm in diameter and weighing 30 mg (with a surface density of 0.04 g/cm^2). The elements were determined via synchrotron radiation X-ray fluorescence analysis (SR XRFA) on the elemental analysis station of the VEPP-3 collector at the Budker Institute of Nuclear Physics' Siberian Center of Synchrotron and Terahertz Radiation. The samples were measured at a 23 keV energy of exciting radiation. Each measurement took 300 to 500 s for weighed amounts of plant matter and soil. The synchrotron radiation was monochromatized using a monochromator based on a silicon crystal of the butterfly type with (111) working planes. The radiation of fluorescence was recorded on a PentaFET detector (Oxford Instruments) with an energy resolution of ~135 eV (at 5.9 keV on the K_α line of Fe). The main characteristics of the experimental station and other aspects of the work were described in [3, 4].

The obtained spectra were processed using the AXIL program. The concentrations of elements were determined according to external standards. Russian standards of grass–grain mixtures (GSO SORM1) and Baikal bottom silt (BIL-1) were used as our reference standards [12]. The value of the error—the reproducibility of the results from analysis—was obtained by making ten parallel measurements according to SORM1 in three specimens of a sample and five according to BIL-1. The limits of detection and relative standard deviation S_r were calculated in the same way (Table 1). We can see from Table 1 that the values of C_{min} varied, depending on the element and the reference standard that was used. A general drop in the limits of detection is noticeable when moving from light elements (K) to heavier elements (Pb).

Enrichment and dispersion of elements for plants growing under urban conditions relative to background conditions were estimated by calculating the local enrichment factor EF_l and dispersion factor DF_l: $EF_l = C_u/C_b$ and $DF_l = C_b/C_u$, where C_b and C_u are the concentrations of the element in the background and urban samples, respectively. To find changes in the chemical composition of the plants under the action of dust–gas emissions in urban habitats, we used a complex parameter (biogeochemical transformation factor Zv [13]) calculated with the formula

$$Zv = \sum_{1}^{n_1} EF_l + \sum_{1}^{n_2} DF_l - (n_1 + n_2 - 1), \quad (1)$$

where n_1 and n_2 are the numbers of elements with $EF_l > 1.5$ and $DF_l > 1.5$, respectively.

RESULTS AND DISCUSSION

A comparative analysis of the soil from the points of collecting our plant specimens showed that the differences between the concentrations of elements were negligible (Table 2). A rise in the concentrations of Ca, As, Br, Sr, and Mo and a drop in that of Mn in the urban soils were excessive, relative to the control samples. Overall, it should be noted that the concentrations of chemical elements in our soil specimens barely exceeded the background levels in the soils of Novosibirsk and Novosibirsk oblast presented in [14–17]. An exception was Br, the concentration of which at the point of collection in the city was six times higher that the background parameters. The concentration of Sr also grew by 30%, due likely to the point of collecting the specimens being near heating plants and motorways. Overall, it should be noted that the pollution with heavy metals at the point of collection under urban conditions was found to be negligible, due likely to the area being in a square away from motorways.

Table 1. Limits of detection C_{min} at a 23 keV energy of excitation, according to Russian standards GSO SORM1 and BIL-1, ppm

Elements	SORM1		BIL-1	
	C_{min}	S_r, %[1]	C_{min}	S_r, %
K	27	10	206	6
Ca	13	11	106	8
Ti	3	26	31	5
V	0.1	19	8	8
Cr	1.9	64	6	6
Mn	0.9	9	8	4
Fe	0.6	8	5	4
Co	0.03	36	0.3	9
Ni	0.3	39	3	4
Cu	0.15	14	2	5
Zn	0.2	12	1	5
As	н.а.[2]	–	1	3
Br	0.05	11	0.4	8
Rb	0.07	11	0.3	9
Sr	0.1	12	0.3	7
Y	0.6	29	0.4	12
Zr	0.5	58	0.3	16
Nb	1.4	40	0.3	9
Mo	0.07	10	0.1	14
Pb	0.05	35	0.5	14

[1] S_r is the relative standard deviation.
[2] N/C: The concentration of the chemical element is not certified.

Our study of the concentrations of macro- and microelements in the above-ground organs of plants showed that the concentration of macroelements K and Ca was higher in leaves than in stems, irrespective of the taxon and habitat (Table 3). The average concentration of K in plants was 1.5 times lower under urban conditions than those of the background. In contrast, the concentration of Ca in the leaves of urban plants grew by 1.7 times. The total concentration of microelements in the plants of the polluted area was higher than in the background area. The greatest differences were found in specimens of the leaves of *M. bracteata* (1258 ppm). The total concentration of microelements in the leaves of *M. bracteata* from the urban area thus grew 2.1 times, relative to the background value. The species were arranged in descending order with respect to the level of element accumulation: *M. bracteata* > *S. media* > *S. chamaedryfolia*. Changes in the total concentration of elements as the anthropogenic load grew were less substantial in the stems of the plants.

It was found that the concentration of Ca, Ti, V, Fe, Co, Br, Sr, Y, Zr, Nb, and Pb grew in the plants subjected to anthropogenic action, while the concentrations of K, Zn, and Mo fell relative to the background. The worst pollution with heavy metals under urban conditions was noted in the plants of *M. bracteata*.

We calculated biogeochemical transformation factor *Zv*, which reflects abnormal ratios of elements in plant bodies as a result of the intensification of anthropogenic loads. The greatest changes in the elemental composition of the plants under anthropogenic action

Table 2. Concentration of elements in the soils from the collection points of the plants under urban and background conditions (ppm per the air-dry weight)

Element	City	Control	Background concentration of elements in the soils, according to the literature [13–16]
K	12685 ± 767[1]	14769 ± 893	–[2]
Ca	44343 ± 3400	15565 ± 1193	–
Ti	2691 ± 133	3482 ± 172	4100[4]
V	55 ± 5	69 ± 6	60[3]
Cr	36 ± 2	41 ± 2	80[3]
Mn	659 ± 26	795 ± 31	750[3]
Fe	22123 ± 988	22836 ± 1020	38000[6]
Co	11 ± 1	9 ± 1	12[3]
Ni	32 ± 1	34 ± 2	35[3]
Cu	27 ± 1	18 ± 1	30[3]
Zn	59 ± 3	46 ± 2	70[3]
As	15 ± 1	4 ± 0	15[3]
Br	22 ± 2	3 ± 0.2	1.2–3.6[5]
Rb	46 ± 4	47 ± 4	–
Sr	223 ± 16	142 ± 10	170[3]
Y	17 ± 2	17 ± 2	–
Zr	177 ± 29	128 ± 21	250[3]
Nb	7 ± 1	7 ± 1	15[3]
Mo	0.7 ± 0.1	0.3 ± 04	3[3]
Pb	15 ± 2	16 ± 2	15[3]

[1] Average value ± standard deviation.
[2] The dashes mean there is no data.
[3] Background concentration of heavy metals in soils of southern West Siberia [14].
[4] Background concentration of heavy metals in soils of southern West Siberia [15].
[5] Background concentration of halogens in soils of West Siberia [13].
[6] Background concentration of elements in soils of Novosibirsk oblast [16].

were noted in plants of the species *M. bracteate*. The *Zv* of its leaves is 65.7, which is 1.7–4.3 times higher than in plants of the genus *Spiraea*. Plants of *S. chamaedryfolia* are the most resistant to anthropogenic pollution (*Zv* = 15.3) (Table 4). Since *S. chamaedryfolia* was identified as the specimen most tolerant to pollution under urban conditions, it can be recommended for use as a standard.

CONCLUSIONS

We have presented data on the concentrations of 20 elements in urban and background soils of Novosibirsk. It was noted that the concentrations of Ca, Br, Sr, Cu, Zn, Zr, and Mo in urban soils exceed the background values.

At least 20 elements were identified in the leaves and stems of two species of plants from the genus *Spiraea*: *S. chamaedryfolia* and *S. media*, in addition to *Myricaria bracteata* growing under the conditions of the urban ecosystem and background conditions. The concentrations of Ca, Ti, V, Fe, Co, Br, Sr, Y, Zr, Nb, and Pb grew in the plants under technogenic loads, while the concentrations of K, Zn, and Mo fell relative to the background. The most severe pollution with heavy metals under urban conditions was noted in the

Table 3. Concentration of elements in the above-ground organs of plants from the genera *Spiraea* and *Myricaria* growing under urban and background conditions in Novosibirsk (ppm per air-dried weight)

Element	Plant organ	City			Control (Central Siberian Botanical Garden, Siberian Branch, Russian Academy of Sciences)		
		1	2	3	1	2	3
K	l	19296 ± 1167[4]	15289 ± 925	13094 ± 792	27678 ± 1674	17576 ± 1063	28509 ± 1724
	s	7960 ± 481	7089 ± 429	7455 ± 451	13941 ± 843	7107 ± 430	11456 ± 693
Ca	l	17517 ± 1343	15195 ± 1165	32896 ± 2522	17142 ± 1314	8726 ± 669	16828 ± 1290
	s	7849 ± 602	15004 ± 1150	4111 ± 315	13351 ± 1024	25509 ± 1956	3929 ± 301
Ti	l	14 ± 1	11 ± 1	32 ± 2	13 ± 1	8 ± 0	5 ± 0
	s	14 ± 1	8 ± 0	4 ± 0	27 ± 1	14 ± 1	8 ± 0
V	l	0.5 ± 0.0	0.4 ± 0.0	0.4 ± 0.0	0.8 ± 0.1	0.5 ± 0.0	0.2 ± 0.0
	s	0.4 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	1.2 ± 0.1	0.8 ± 0.1	0.3 ± 0.0
Cr	l	NDA[5]	1.7 ± 0.1	3.1 ± 0.2	NDA	0.1 ± 0.0	4.0 ± 0.2
	s	NDA	0.8 ± 0.05	1.8 ± 0.1	NDA	NDA	1.7 ± 0.1
Mn	l	63 ± 2	92 ± 4	94 ± 4	103 ± 4.0	65 ± 3	158 ± 6
	s	38 ± 2	104 ± 4	18 ± 1	72 ± 2.8	108 ± 4.3	34 ± 1.3
Fe	l	268 ± 12	222 ± 10	739 ± 33	129 ± 5.7	87 ± 4	192 ± 9
	s	200 ± 9	112 ± 5	83 ± 4	125 ± 5.6	68 ± 3.0	52 ± 2.3
Co	l	0.1 ± 0.0	0.1 ± 0.0	0.3 ± 0.0	BLD[6]	BLD	0.1 ± 0
	s	0.1 ± 0.0	BLD	0.1 ± 0.0	BLD	BLD	BLD
Ni	l	1.3 ± 0.1	1.3 ± 0.1	1.2 ± 0.1	1.9 ± 0.1	1.2 ± 0.1	1.1 ± 0.1
	s	0.9 ± 0.0	1.1 ± 0.1	2.5 ± 0.1	1.5 ± 0.1	0.9 ± 0.0	2.2 ± 0.1
Cu	l	4.8 ± 0.3	5.5 ± 0.3	5.2 ± 0.3	6.0 ± 0.3	4.5 ± 0.2	4.8 ± 0
	s	4.1 ± 0.2	4.6 ± 0.2	6.2 ± 0.3	10.5 ± 0.5	6.3 ± 0.3	5.2 ± 0.3
Zn	l	13 ± 1	24 ± 1	36 ± 2	16 ± 1	39 ± 2	63 ± 3
	s	67 ± 3	45 ± 2	23 ± 1	73 ± 4	121 ± 6	34 ± 2
As	l	0.1 ± 0.0	1.0 ± 0.0	NDA	0.5 ± 0.0	0.4 ± 0	NDA
	s	0.7 ± 0.0	0.4 ± 0.0	1.4 ± 0.04	0.9 ± 0.0	1.0 ± 0.0	0.2 ± 0.0
Br	l	2.1 ± 0.2	0.9 ± 0.1	37.8 ± 2.9	6.0 ± 0.5	1.5 ± 0.1	20.8 ± 1.6
	s	0.4 ± 0.0	0.5 ± 0.0	35.1 ± 2.7	2.0 ± 0.2	0.4 ± 0.0	12.3 ± 0.9
Rb	l	5 ± 0	3 ± 0	9 ± 1	5 ± 0	2 ± 0	8 ± 1
	s	3 ± 0	2 ± 0	3 ± 0	3 ± 0	1 ± 0	2 ± 0
Sr	l	141 ± 10	93 ± 7	274 ± 20	104 ± 7.5	43 ± 3	121 ± 9
	s	100 ± 7	149 ± 11	64 ± 5	116 ± 8	144 ± 10	46 ± 3
Y	l	0.1 ± 0.0	0.6 ± 0.1	10.1 ± 1.2	0.1 ± 0.0	NDA	2.2 ± 0.3
	s	0.9 ± 0.1	0.9 ± 0.1	0.2 ± 0.0	NDA	0.3 ± 0.0	0.3 ± 0.0
Zr	l	0.8 ± 0.1	0.6 ± 0.1	9.1 ± 1.5	0.5 ± 0.1	0.3 ± 0.0	0.9 ± 0.2
	s	0.9 ± 0.1	0.6 ± 0.1	0.4 ± 0.1	0.6 ± 0.1	0.5 ± 0.1	0.4 ± 0.1
Nb	l	0.6 ± 0.1	0.3 ± 0.0	2.3 ± 0.2	0.3 ± 0.0	0.9 ± 0	0.1 ± 0.0
	s	1.9 ± 0.2	0.1 ± 0.0	1.9 ± 0.2	1.0 ± 0.1	NDA	1.2 ± 0.1
Mo	l	0.7 ± 0.1	0.3 ± 0.0	1.9 ± 0.3	2.0 ± 0.3	4.3 ± 0.6	3.6 ± 0.5
	s	0.3 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.8 ± 0.1	1.6 ± 0.2	0.4 ± 0.1
Pb	l	1.0 ± 0.1	1.7 ± 0.2	2.9 ± 0.4	1.1 ± 0.2	0.9 ± 0.1	1.5 ± 0.2
		1.8 ± 0.2	1.0 ± 0.0	2.0 ± 0.3	1.4 ± 0.2	1.3 ± 0.2	0.8 ± 0.1

[1] *Spiraea chamaedryfolia*.
[2] *Spiraea media*.
[3] *Myricaria bracteata*.
[4] Average value ± standard deviation.
[5] NDA, no data available.
[6] BLD, below the limit of detection (0.01 ppm).

Table 4. Environmental state of plants from the genera *Spiraea* and *Myricaria* growing in Novosibirsk

Species	*Zv*
Spiraea chamaedryfolia	15.3
Spiraea media	38.4
Myricaria bracteata	65.7

plants of *M. bracteata*. Higher values of biogeochemical transformation factor *Zv* were recorded for the leaves of *M. bracteata*, indicating more substantial changes in the microelemental composition compared to *Spiraea*. *S. chamaedryfolia*, identified as the specimen most tolerant to pollution under urban conditions, can be recommended for use as a standard and in green construction. The species *M. bracteata* is recommended as a bioindicator plant for environmental pollution.

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CONFLICT OF INTEREST

The authors declare they have no conflicts of interest.

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