

# ANALYSIS OF TRACE ELEMENT ATMOSPHERIC DEPOSITION BY BARBULA INDICA MOSS AT BAOLOC USING THE TOTAL REFLECTION X-RAY FLUORESCENCE TECHNIQUE

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## Abstract

*In this investigation, the Total Reflection X-ray Fluorescence (TXRF) technique detected 24 elements: Al, P, S, Cl, K, Sr, Sc, Ti, Mn, Fe, Co, Cu, Zn, As, Br, Ba, La, Eu, Tb, Dy, Ta, Pb, Th, and U in Barbula indica moss collected at Baoloc (Vietnam) from November 2019 to March 2020. Factor analysis was used to explain contamination sources at the sampling sites. This study showed that the passive moss biomonitoring and TXRF techniques are efficient and very suitable for detecting trace elements due to atmospheric deposition in developing countries, especially Vietnam and some Asian countries.*

**Keywords:** Atmospheric deposition; Baoloc; Barbula indica; TXRF.

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# PHÂN TÍCH NGUYÊN TỐ VẾT LẮNG ĐỘNG TRONG KHÔNG KHÍ QUA RÊU *BARBULA INDICA* TẠI THÀNH PHỐ BẢO LỘC SỬ DỤNG KỸ THUẬT HUỖNH QUANG TIA X PHẢN XẠ TOÀN PHẦN

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## Tóm tắt

Trong nghiên cứu này, kỹ thuật huỳnh quang tia X phản xạ toàn phần (TXRF) được ứng dụng đã xác định được 24 nguyên tố, bao gồm: Al, P, S, Cl, K, Sr, Sc, Ti, Mn, Fe, Co, Cu, Zn, As, Br, Ba, La, Eu, Tb, Dy, Ta, Pb, Th, và U trên rêu *Barbula Indica* tại thành phố Bảo Lộc (Việt Nam) từ tháng mười một năm 2019 đến tháng ba năm 2020. Kết quả cũng đã dự đoán những nguồn ô nhiễm mang lại. Ở nghiên cứu này cho thấy việc sử dụng mẫu rêu có sẵn, và kỹ thuật TXRF là hiệu quả, rất thuận lợi để xác định sự lắng đọng các nguyên tố vết trong không khí cho những quốc gia đang phát triển, đặc biệt là Việt Nam và các nước Châu Á.

**Từ khóa:** Bảo Lộc; Rêu *Barbula indica*; Sự lắng đọng không khí; TXRF.

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## 1. INTRODUCTION

Today, air pollution is a serious problem in developing countries, including Vietnam. Air pollution is a result of industrialization and urbanization. The main sources of environmental pollutants are development industry, mineral processing, farming, and transport activity. Numerous studies using various moss species as indicators for environmental pollution have been performed (Rühling & Tyler, 1968, 1969, 1970). Moss acts as a biomonitoring station to detect multi-elemental atmospheric deposition. The advantages of this method include easy implementation and low cost. Therefore, it is affordable for developing countries.

The moss transplant technique was introduced by Gjengedal and Steinnes (1990), who applied moss to determine trace elements in the air. Commonly, there are two moss techniques: native species and active biomonitoring. Sucharová and Suchara (1998) used native species in assessing temporal or spatial changes in atmospheric deposition of trace elements in Bryophytes mosses. They determined the atmospheric loads of 13 elements (Al, As, Cd, Co, Cr, Cu, Fe, Mo, Ni, Pb, S, V, and Zn). Mosses were also used for active biomonitoring with wet and dry moss bags to examine trace element atmospheric deposition. The moss bag technique is a simple, cost-effective, and eco-friendly tool for air monitoring. Fernández and Carballeira (2000) have used transplanted mosses of the species *Scleropodium purum* to detect levels of Co, Cr, Cu, K, Ni, Pb, Se, and Zn.

Most applications of the moss technique have been widely used for metal deposition monitoring in Europe. The moss species that have been used include *Pleurozium schreber*, *Hylocomium splendens*, *Scleropodium purum*, and *Hypnum cupressiforme* (Frontasyeva et al., 2004; Frontasyeva and Harmens, 2019; Harmens et al., 2010). These mosses are rarely found in Asia; instead, other moss types, such as *Hypnum plumaeforme*, *Taxithelium instratum*, *Thuidium tamariscellum*, and *Barbula indica* were used (Abdullah, Saat, & Hamzah, 2012; Doan Phan, Trinh, Khiem, Frontasyeva, & Quyet, 2019; Khiem et al., 2020; Lee, Li, Zhang, Peng, & Zhang, 2005). Neutron activation analysis, inductively coupled plasma mass spectrometry, and energy dispersive X-ray fluorescence are multi-element analytical techniques that provide quantification at low levels and have been used to analyze trace elements in moss samples. In this study, we focused on the analysis of elements in moss using the total reflection X-ray fluorescence (TXRF) technique, which offers even better detection limits than other widely used multi-element detection techniques.

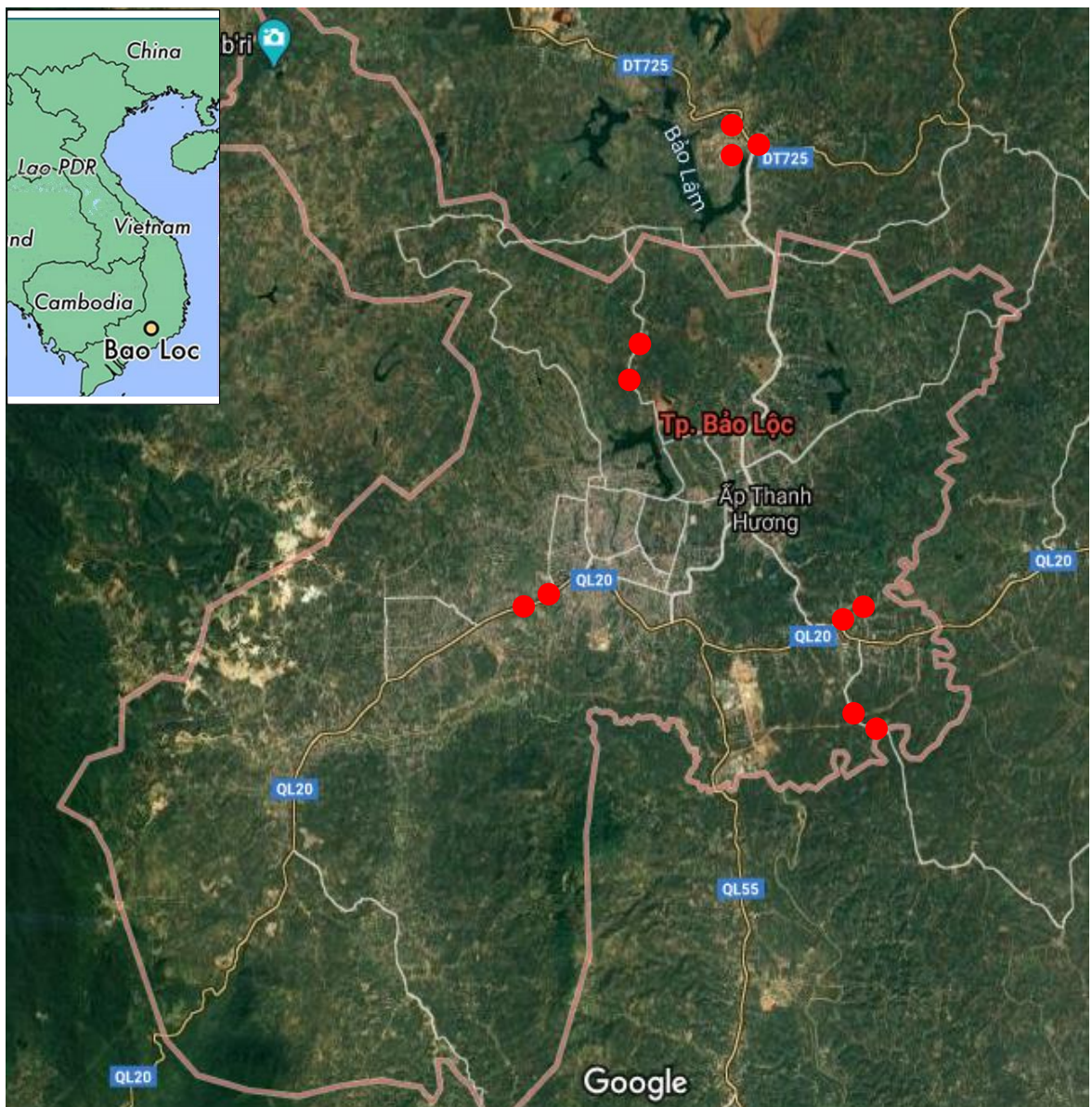
## 2. MATERIALS AND METHODS

### 2.1. Sampling areas

Baoloc, the second largest city of Lamdong province in the Central Highlands region of Vietnam, is located at 11°32'52.73"N latitude and 107°48'27.79"E longitude. It covers an area of around 232.56 km<sup>2</sup> and lies 846 m above sea level. Baoloc's climate is classified as tropical with an average temperature of 21°C. The rainfall here is about

2480 mm per year. Normally, Baoloc has two seasons: The dry season lasts six months from November to May, and the wet season lasts from May to November.

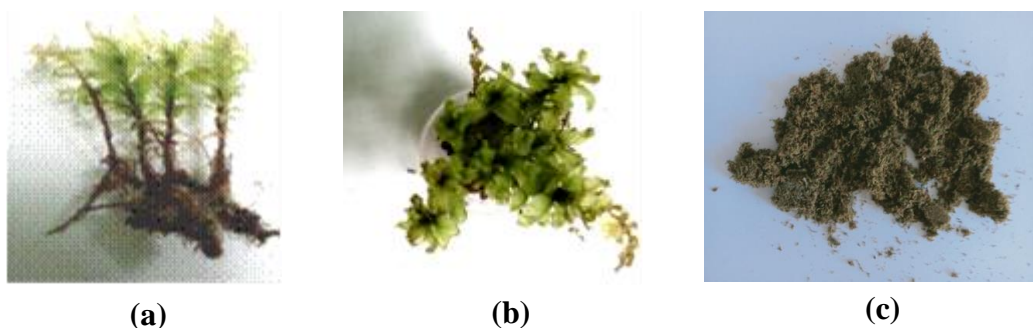
In this work, *Barbula indica* was chosen to study the atmospheric deposition of trace elements. The moss samples were collected at the end of the rainy season from November 2019 to March 2020 at 11 places in Baoloc (Fig. 1). The sampling points were assumed to have different pollution levels due to various anthropogenic activities, such as roads, farms, and the Tan Rai alumina refinery.



**Figure 1. Sampling sites in Baoloc**

## 2.2. Preparing moss samples

The *Barbula indica* moss morphology and a raw moss sample are shown in Fig. 2. To minimize the influence of the substrate, moss samples were collected from the tree at least 1.5 m above the ground and only the top, green part was used for analysis. The collected moss samples were cleaned of soil particles, washed three times with distilled water, and then dried at 40 °C for 50 h. The dried moss was crushed and homogenized to a moss powder (~0.5 mm) using an analytical sieve shaker AS 300 control for 30 minutes, followed by milling to a size smaller than 50 µm with a Retsch mixer mill MM 400.



**Figure 2. Side- and overhead-views of *Barbula indica* moss (a, b) and raw moss sample (c)**

An amount of 0.5 g of fine moss powder was placed in a digestion vessel. After adding 10 mL of concentrated HNO<sub>3</sub>, the sample was gently swirled, then left at room temperature until the evaporation of nitrogen dioxide had ceased. The vessel was placed into a microwave digestion system (MARS6) which has three periods of digestion: first, the temperature inside the vessel was increased up to 220 °C in 15 min; second, the sample was held at that temperature for 50 min; finally, it was cooled to room temperature.

When the digestion was completed, the digested solution was diluted to 10 mL with distilled water. An aliquot of 1.35 mL sample was transferred to a polymer container and added to 0.15 mL gallium internal standard liquid (10 ppm) until the sample reached 1 ppm gallium. The sample must be thoroughly homogenized by an automatic sample shaker. After thorough homogenization, 10 µl of the sample were transferred to a sample carrier and then dried at 30 °C. Fig. 3 shows the TXRF sample.



**Figure 3. Liquid moss sample on the sample carrier (a) and dry moss sample (b)**

### 2.3. TXRF technique

The characteristic X-ray spectrum for each moss sample was determined with an S2 PICOFOX™ TXRF spectrometer operated at 50 kV high voltage and a maximum tube rating of 50 W. All 11 moss samples and the gain correction sample were placed into the sample changer (Fig. 4). The TXRF spectrometer characteristics are described in detail in Towett, Shepherd, and Cadisch (2013).



**Figure 4. Sample transfer to the TXRF spectrometer**

The necessary reset of the spectroscopic amplification is made with the gain correction software function. In this process, a correction value is transferred to the spectroscopic amplifier after a reference measurement having a known fluorescence peak. For the gain correction, a suitable as mono-element standard sample was used. The measurement time of 120 s for each sample, sufficient for the necessary statistics, was established.

The quality of the results obtained with the TXRF technique is greatly impacted by the accuracy of the instrument sensitivity curve. The sensitivity is determined by the measurement of a Kraft sample (having 1 ppm of all 10 elements: Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Rb), for which the fluorescence intensity, standardized on mass, time, and tube current, is detected by the instrument. The fit quality is a statistical parameter reflecting the quality of the deconvolution. For all channels, the standardized sum of the squares of the differences between the measured and calculated deconvoluted intensities is calculated. The value of the fit quality should preferably be less than 10. High values (>10) are an indication of misidentified or unidentified elements, respectively, or inaccurate gain correction. The fit quality function is  $\sum_{i=n_1}^{n_2} \frac{1}{\delta_i^2} (y_{i+1} - y_i)^2$  where  $n_1$  is the first channel of peak  $i$  (the left channel),  $n_2$  is the last channel of peak  $i$  (the right channel),  $y_{i+1}$  is the number of counts for channel  $i+1$ ,  $y_i$  is the number of counts for channel  $i$ , and  $\delta_i = \sqrt{N_i + 2N_{BG}}$  where  $\delta_i$  is the standard deviation for the peak area,  $N_i$  is the net peak area for element  $i$ , and  $N_{BG}$  is the background area.

### 3. RESULTS AND DISCUSSIONS

Up to 24 elements, including Al, P, S, Cl, K, Sr, Sc, Ti, Mn, Fe, Co, Cu, Zn, As, Br, Ba, La, Eu, Tb, Dy, Ta, Pb, Th, and U, were detected with the TXRF technique in moss samples collected at 11 locations in Baoloc. The trace element concentrations in the

mass samples are presented in Table 1, for which the errors in the concentrations are less than 10%.

**Table 1. The concentration of trace elements in moss samples (in mg.kg<sup>-1</sup>)**

El	Site										
	BL01	BL02	BL03	BL04	BL05	BL06	BL07	BL08	BL09	BL10	BL11
Al	2157.00	2154.00	2156.00	2156.00	4820.00	4581.00	4687.00	2320.00	1472.00	1455.00	1455.00
P	503.00	506.00	505.00	505.00	1048.00	839.00	591.00	803.00	978.00	1014.00	1014.00
S	689.00	678.00	684.00	684.00	2715.00	2568.00	2678.00	682.00	1154.00	1240.00	1240.00
Cl	767.00	741.00	754.00	754.00	927.00	857.00	987.00	750.00	897.00	970.00	970.00
K	2512.00	2478.00	2495.00	2495.00	2926.00	3540.00	3320.00	2489.00	8102.00	8199.00	8199.00
Sr	6.93	6.21	6.57	5.98	6.33	5.42	5.70	6.25	5.20	11.79	11.79
Sc	0.12	0.98	0.55	0.75	2.17	2.43	1.95	nd	nd	nd	0.86
Ti	143.00	168.00	156.00	156.00	320.00	350.00	340.00	147.00	47.00	46.00	46.00
Mn	46.00	42.00	44.00	44.00	70.00	74.00	82.00	39.00	68.00	74.00	74.00
Fe	924.00	911.00	918.00	918.00	5400.00	5064.00	5321.00	841.00	2540.00	2625.00	2625.00
Co	nd	0.34	nd	nd	6.21	5.46	4.72	nd	2.50	2.35	2.35
Cu	7.18	10.60	8.89	8.42	12.50	15.40	9.24	8.47	15.40	16.63	16.63
Zn	57.00	52.00	54.00	47.00	214.00	176.00	87.00	51.00	689.00	746.00	746.00
As	2.46	2.33	2.40	2.40	5.73	5.92	6.45	2.37	0.55	0.52	0.52
Br	2.73	1.97	2.35	2.35	3.42	4.12	3.72	2.22	2.78	2.47	2.47
Ba	18.64	12.56	15.60	15.60	26.58	22.78	23.47	14.59	4.58	5.18	5.18
La	3.58	2.45	3.02	3.02	14.78	17.89	23.96	2.83	1.02	1.36	1.36
Eu	nd	nd	nd	0.34	5.45	6.54	5.89	nd	0.25	0.20	0.20
Tb	nd	nd	nd	0.27	4.32	4.87	5.12	nd	nd	nd	0.47
Dy	0.18	0.23	0.21	0.21	3.21	4.27	3.87	nd	0.26	0.29	0.29
Ta	nd	nd	nd	0.78	4.21	4.52	5.15	nd	nd	0.45	0.37
Pb	2.74	3.54	3.14	3.14	6.54	6.87	5.98	3.27	17.63	18.03	18.03
Th	0.26	0.87	0.57	0.57	2.14	1.87	1.96	0.67	nd	nd	0.34
U	0.77	0.73	0.75	nd	3.21	2.85	2.49	nd	0.45	0.74	0.74

Note: El–element

The mean concentrations of the elements in the moss samples from Baoloc decreased as: K > Al > Fe > S > Cl > P > Zn > Ti > Mn > Ba > Cu > Pb > Sr > La > As > Br > Co > Eu > Ta > Tb > Dy > U > Sc > Th.

Contamination factor (CF) scales were used to determine the contamination levels of each element in the sample (Fernández & Carballeira, 2001). The CF was calculated

as the ratio of the mean value of each heavy metal in a moss sample to the background level, as in the equation (Hakanson, 1980):  $CF_i = \frac{C_i}{BG_i}$  where  $C_i$  is the mean value of the  $i^{th}$  element from the investigated area, and  $BG_i$  is the average value of the three sample sites which have the lowest concentration of the corresponding metal from the investigated area.

The CF values comprise six levels:  $CF < 1$ : No contamination,  $1 < CF \leq 2$ : Suspected contamination,  $2 < CF \leq 3.5$ : Slight contamination,  $3.5 < CF \leq 8$ : Moderate contamination,  $8 < CF \leq 27$ : Serious contamination, and  $27 < CF$ : Extreme contamination (Fernández & Carballeira, 2001). Table 2 shows the contamination factors for 24 elements.

**Table 2. The contamination factors of trace elements in the moss samples**

El	AL	P	S	Cl	K	Sr	Sc	Ti	Mn	Fe	Co	Cu
CF	1.8	1.5	2.0	1.1	1.7	1.3	1.9	3.8	1.4	2.9	1.3	1.5
El	Zn	As	Br	Ba	La	Eu	Tb	Dy	Ta	Pb	Th	U
CF	5.3	5.4	1.3	3.0	5.5	7.9	0.8	5.9	2.6	2.7	2.2	1.8

A comparison of this result with those obtained in previous studies of *Barbula indica* moss in Vietnam and moss in European countries (Barandovski, Stafilov, Sajn, Frontasyeva, & Baceva, 2012) was carried out. The results of the comparison are listed in Table 3.

**Table 3. Comparison of the mean trace element concentrations from atmospheric deposition on *Barbula indica* moss at Baoloc with some previous work (in  $\text{mg}\cdot\text{kg}^{-1}$ )**

Element	Baoloc city our work	Doan Phan et al., 2019			Khiem et al., 2020	Barandovski et al., 2012
		Hue city	Hoian city	Hochiminh city	Hanoi capital	
Na		620.00	1310.00	930.00		
Mg		1550.00	1620.00	1290.00	3866.66	1900.00
Al	2674.00	5800.00	3200.00	4800.00	10591.19	1900.00
Si					39595.76	
P	755.00				1100.14	1100.00
S	1365.00				3238.92	
Cl	852.00	1700.00	2100.00	780.00	1711.59	
K	4250.00	16000.00	17000.00	12000.00	14401.56	4600.00
Sr	7.11					



**Table 3. Comparison of the mean trace element concentrations from atmospheric deposition on *Barbula indica* moss at Baoloc with some previous work (in mg.kg<sup>-1</sup>) (cont.)**

Element	Baoloc city our work	Doan Phan et al., 2019			Khiem et al., 2020	Barandovski et al., 2012
		Hue city	Hoian city	Hochiminh City	Hanoi Capital	
Sc	0.89	1.09	0.80	1.81		
Ti	174.00	271.00	205.00	524.00	691.60	
V		12.20	5.11	8.08		3.50
Cr		11.00	6.80	19.90	26.73	3.50
Mn	60.00	74.00	88.00	77.00	170.95	130.00
Fe	2553.00	3720.00	4810.00	5430.00	6025.18	1500.00
Co	2.18	1.40	1.01	3.28		
Ni		4.20	2.70	9.50	4.42	3.50
Cu	11.76				27.20	3.50
Zn	265.00	126.00	254.00	178.00	397.53	20.00
As	2.88	2.30	3.00	4.10	16.11	
Se		1.40	0.70	0.39		
Br	2.78	10.40	7.70	12.80	36.11	
Kr						
Rb					151.17	
Sb		1.40	0.89	1.00		
Cs		1.58	1.18	5.30		
Ba	14.98	58.00	83.00	101.00	1545.55	34.00
La	6.84	3.10	2.24	5.60		
Ce		6.20	4.20	11.70		
Sm		0.51	0.35	1.15		
Eu	1.72					
Tb	1.37					
Dy	1.18					
Ta	1.41	0.09	0.07	0.15		
Pb	8.08				231.55	4.60
Th	0.84	1.78	0.86	1.49		
U	1.16	0.62	0.23	1.10		

For Hue city, Hoian city, and Hochiminh City, the element concentrations are near the values found in our work and some are slightly higher. For Hanoi, all element

concentrations are higher than in Baoloc. The concentrations of Al, P, S, Cl, K, Ti, Mn, Fe, Cu, Zn, As, Br, Ba, and Pb in Hanoi are higher than in Baoloc: 4.0, 1.5, 2.4, 2.0, 3.4, 4.0, 2.8, 2.4, 2.3, 1.5, 5.6, 13.0, 103, and 28.7, respectively, especially for barium and lead.

According to Pacyna and Pacyna (2002) and Cucu-Man, Mocanu, Culicov, Steinnes, and Frontasyeva (2004), the main pollution sources in our study area can be explained as follows:

- *Sample sites*: BL01, BL02, BL03, and BL04 are major roads where traffic activity is high. In particular, BL01 and BL02 are at the entrance of Baoloc pass; BL03 and BL04 are in the city center of Baoloc; BL10 and BL11 are in the area between Baoloc city and Dilinh district (along 20<sup>th</sup> road); BL08, and BL09 are the places where silk is produced. Notably, the pollution in these places is affected by windblown dust and traffic emissions, especially gasoline-burning vehicles, cars, and motorbikes.
- *Three special sample sites*: B05, B06, and B07 are near the Tan Rai alumina refinery. All concentrations of trace elements, especially aluminum, are higher there than at other moss sample sites in this work. That suggests the pollution at these sites was effected by aluminum ore processing.

#### 4. CONCLUSIONS

In this investigation, we applied the TXRF technique to determine trace element concentrations from atmospheric deposits on *Barbula indica* moss samples and to estimate the metal pollution in Baoloc. The TXRF technique is useful and suitable. The result is expressed through the number of analytical elements. A total of 24 elements were detected, including Al, P, S, Cl, K, Sr, Sc, Ti, Mn, Fe, Co, Cu, Zn, As, Br, Ba, La, Eu, Tb, Dy, Ta, Pb, Th, and U. We compared our data with some previous research on trace element atmospheric deposition on *Barbula indica* moss in Vietnam, and the results show that the element concentrations at Baoloc are lower than other locales, although a little higher than the concentrations found by Barandovski et al. (2012). Most of the elements from atmospheric deposits in Baoloc are at the “suspected” to “slight” contamination level. The air pollution sources of these elements are possibly due to traffic and aluminum ore processing.

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