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Aluminum determination by instrumental neutron activation analysis in tree barks

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Abstract Neutron activation analysis (NAA) for Al determination in tree bark samples containing phosphorus and silicon was studied. The contributions of the $^{31}P(n,\alpha)^{28}Al$ and $^{28}Si(n,p)^{28}Al$ reactions to the $^{27}Al(n,\gamma)^{28}Al$ reaction were corrected determining interference correction factors. Analytical quality control was evaluated by analyzing certified reference materials. The results presented good accuracy and precision. Replicate Al determinations in bark samples showed relative standard deviations ranging from 4.2 to 10.5%. The detection limits were lower than the mass fractions found in the samples, demonstrating the possibility to apply NAA in Al determinations in tree barks for biomonitoring studies and purposes.

Keywords Aluminum · Neutron activation analysis · Tree barks · Interferences

Introduction

Over the years, tree barks have been widely analyzed to evaluate environmental pollution and several elements have been determined in this biomonitor. However, Al determinations in tree barks for environmental monitoring purposes are very scarce. This element is toxic under special circumstances and is considered a harmful contaminant to human health and can be found throughout the environment [1]. Thus, Al determinations in environmental samples are of great interest. Its evaluation as an atmospheric pollutant has received considerable attention. Aluminum is present in the atmosphere as particulate matter or fine particles originating from natural soil erosion, agricultural, mining and industrial activities, volcanic eruptions or coal combustion. This element has been claimed to develop neurodegenerative diseases such as Alzheimer and amyotrophic lateral sclerosis [2].

Several analytical techniques have been proposed for Al determinations such as inductively coupled plasma mass spectrometry (ICP MS) [3] inductively coupled plasma atomic emisson spectrometry (ICP AES) [3, 4], spectrofluorimetric method [5], liquid chromatography with a detection by the atomic absorption spectrometry with flame atomization (HPLC FAAS) [6] and neutron activation analysis (NAA) [7–10]. Among these methods, NAA is a non-destructive method and free from blank reagents besides requiring minimum sample preparation that avoids contamination. Furthermore this method presents high sensitivity has been also applied in the Al determinations in tree barks [10–12].

In INAA, this element is determined by measuring the peak at 1778.99 keV gamma ray of ^{28}Al with half life of 2.24 min formed in the reaction $^{27}Al(n,\gamma)^{28}Al$. Unfortunately the interference of Al determination by INAA is due to P and Si, which form ^{28}Al by the reactions $^{31}P(n,\alpha)^{28}Al$ and $^{28}Si(n,p)^{28}Al$. The contribution of these interferences can not be neglected depending on the relation between thermal, epithermal and fast neutron fluxes as well as the relation between the mass fractions of P and Al and of Si and Al in the sample.

In this study Al determination was carried out by INAA in tree bark samples and in certified reference materials.

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The interference corrections were accomplished by determining correction factors and using published or determined mass fraction data of P and Si. The Si mass fractions were determined by epithermal neutron activation analysis (ENAA) by measuring gamma ray energy of 1273.36 keV of ^{29}Al formed in the reaction ^{29}Si(n,p)^{29}Al and P determined by INAA by measuring beta activity of 1710 keV of ^{32}P formed in the reaction ^{31}P(n,γ)^{32}P.

Experimental

Certified reference materials and tree bark samples

The following certified reference materials (CRMs) CTA-FFA-1 Fine Fly Ash, INCT-MPH-2 Mixed Polish Herbs, INCT-TL-1 Tea Leaves, NIST 1515 Apple Leaves, NIST 1566b Oyster Tissue, NIST 1570a Trace Elements in Spinach Leaves, NIST SRM Tomato Leaves, NIST 1633b Constituent Elements in Coal Fly Ash and NIST 1646a Estuarine Sediment were used for analytical quality control of Al determinations. In order to obtain CRMs results on dried basis, the percentages of moisture of these materials were determined by drying aliquots of each material according to its respective certificates. The percentages of loss obtained in this process for each material were used to calculate mass fractions of the element on dry basis.

Samples of tree barks were collected from different arboreal species *Ligustrum vulgare* of the Oleaceae family and *Tipuana tipu* and *Poicianella pluviosa,* both belonging to the Fabaceae family. The bark of these species are commonly used as biomonitor of air pollutants [13–15]. The sampling sites were in São Paulo city (latitude 24°29′15″S, longitude 47°50′37″W) and in two cities of the interior of the State of São Paulo: Presidente Prudente (latitude 22°07′32″S, longitude 51°23′20″W) and Marilia (latitude 24°29′15″S, longitude 49°56′45″W) located 559 and 444 km respectively from São Paulo city. According to the Environmental Company of the State of São Paulo (CETESB) [16, 17] the particulate matter averages measured in Presidente Prudente and Marilia during the period from 2010 to 2016 were lower than those measured in the city of São Paulo.

The barks were collected and prepared for the analyses according to Moreira et al. [13]. Briefly, the procedure consisted of removing the bark samples from the trees at chest height of about 1.5 m from the topsoil level and placed into paper bags. According to Schelle et al. [18] contamination from soil is limited to 1.5 m of the trunk. If the samples were wet, they were dried in an oven with air circulation at 40 °C. The bark surface layer was cleaned using a nylon toothbrush and then 3 mm of the outer layer was removed using a Ti grater. This sample was ground for homogenization using an agate-type ball mill (Fritsch, Pulverisette 0) and, finally stored in a desiccator.

Preparation of synthetic standards of the elements Al, P and Si

Synthetic standards of the elements Al, P and Si were prepared using certified stock standards provided from Spex CertiPrep, USA. These solutions were pipetted on sheets of Whatman No. 40 filter paper, dried inside a dessicator and sealed in polyethylene envelopes previously cleaned. According to the certificates of the solutions, the impurity of Al in the P standard solution was low (<0.04 mg L^{-1}) and in Si solution, 60 mg L^{-1}. The reagent ammonium dihydrogen phosphate, Puratronic of 99.998% purity from Alfa Aesar weighed in polyethylene envelope was also used as P standard in addition to the synthetic standard. For Si standard, pieces of metallic silicon of 99.999% purity from Alfa Aesar was also used as standard this element.

Experimental procedure for Al determinations by neutron activation analysis

An aliquot of about 180 mg of each CRM or bark tree sample weighted in a polyethylene envelope was irradiated along with standard of Al and P or Si at the IEA-R1 nuclear research rector using pneumatic transfer system for short irradiations. The irradiation times varied from 5 to 30 s depending on the sample. The thermal, epithermal and fast neutron fluxes were (in n cm^{-2} s^{-1}) 1.9×10^{12}, 5.4×10^{10} and 3.7×10^{11}, respectively in the irradiation position. The decay time of about 3 min was required to mount the irradiated samples and standards in planchets. For counting the sample and standards were measured in the same geometry. The counting system consisted of a hyperpure Ge detector Model GC3020 coupled to a Digital Spectrum Processor DSA 1000, both from Canberra. The resolution (FWHM) of the system was 0.90 keV for 122 keV gamma-ray peak of ^{57}Co and 1.87 keV for 1332 keV gamma ray peak of ^{60}Co. The counting time used was of 200 s. Spectra were collected and processed using Canberra Genie 2000 Version 3.1 software. The peak of 1778.99 keV of ^{28}Al and half-life of 2.24 min was used to identify ^{28}Al. The mass fraction of the element was calculated by comparative method [19].

Determination of interference contributions of P and Si in the determination of Al

These interference contributions were determined using interference correction factors for P and Si obtained experimentally, and knowing the mass fractions of P and Si in the sample. These correction factors were obtained irradiating standards of Al and P or Si and measuring the counting rates of ^{28}Al. The correction factor for each interfering element was calculated using the relation [20].

$$F = \left[A_{sp(n,\gamma)}\right] / \left[A_{sp}\right] \tag{1}$$

where F is the correction factor, $A_{sp(n,\ \gamma)}$ is the specific activity of ^{28}Al produced by the reaction ^{27}Al(n,γ)^{28}Al and A_{sp} is the specific activity of ^{28}Al produced by the interference. The specific activities must be corrected for same decay time. Once the correction factors for P and Si were determined, the corrected mass fraction of Al was calculated using the following expression

$$C_{\text{corrected}} = C_{\text{apparent}} - [(F_{\text{P}} \times C_{\text{P}}) + (F_{\text{Si}} \times C_{\text{Si}})] \tag{2}$$

where $C_{\text{corrected}}$ is the true mass fraction of Al, C_{apparent} is the apparent or uncorrected mass fraction of Al, F_{P} and F_{Si} are the correction factors for P and Si respectively and C_{P} and C_{Si} are the mass fractions of P and Si respectively in the sample.

Mass fractions of phosphorus in the certified reference materials and tree bark samples

Phosphorus mass fractions in the samples are needed to calculate the interference contribution using relation (2). In this study P mass fractions were determined experimentally or published values were used. Phosphorus was determined by neutron activation analysis irradiating the sample and P standard for 4 or 60 min and after a decay time of about 10 days beta activity of ^{32}P (1710 keV and half-life of 14.28 days) was measured. A Geiger Müller counter was used to measure beta activity and the identification of ^{32}P was performed by counting the sample in different decay times and determining the half-life of ^{32}P. After a decay time of about 10 days gamma activities of the radionuclides formed in the irradiation were not present in the gamma spectra.

Mass fractions of silicon in the certified reference materials and tree bark samples

Silicon in the certified reference materials and in tree bark samples was determined by ENAA and measuring gamma ray energy of 1273.36 keV of ^{29}Al with half-life of 6.56 min formed in the reaction ^{29}Si(n,p)^{29}Al. A sample and the Si standard were irradiated from 20 to 60 s inside a cadmium capsule. The use of covered activation to cut thermal neutrons reduced activation for some elements with high cross sections for thermal neutrons such as Al, Cl and Na. Silicon mass fractions were calculated by comparative method.

Results and discussion

Preliminary experiments showed that the Al, P and Si impurities present in polyethylene envelopes used in the irradiations and in sheets of Whatman filter paper used in the preparation of synthetic standards may be considered negligible. The elements P and Si were not detected and Al was present in these materials in very low quantities. The Al mass found were of (0.14 ± 0.03) and (0.12 ± 0.02) µg for each envelope and sheet of filter paper, respectively.

In the determinations of interference correction factors for Al determination, the following mean values were obtained: $F_{\text{P}} = (2.38 \pm 0.03)$ mg Al/g P and $F_{\text{Si}} = (5.29 \pm 0.03)$ mg Al/g Si in 16 irradiations carried out on different days or times of the day in a same irradiation position of the nuclear reactor. A comparison of our results with literature values presented in Table 1 indicates that the results obtained in this study are within the reported values and this difference could be due to nuclear reactor parameters such as the relations between thermal, epithermal and rapid neutrons fluxes. Therefore, in order to use precise and accurate interference correction factors, they were determined for each irradiation of the sample.

Results of P and Si determinations in the CRMs (Table 2) presented standardized differences [22] or |Z-score| ≤ 1.4 indicating a good agreement of our data with the certified values. There is no certified value for P in NIST SRM 1566b Oyster tissue as well as for Si in INCT MPH-2 Mixed Polish Herbs. The results obtained for P are systematically above certified ones. A possible explanation for these results would be the interference in the counts of ^{32}P. These measurements for P determinations were carried out using a Geiger Muller counter and the ^{32}P radioisotope was identified by determining the half-life in all samples. However, gamma and beta activities of other radioisotopes of long half-lives could be contributing in the ^{32}P measurements. The P and Si results obtained in this study were used to calculate the interference contributions of these elements in the Al determination.

Results for P and Si in tree bark samples presented in Table 3 indicate that their mass fractions depend on the sample, since they were collected in sampling sites with different pollution levels and from different species of trees. The detection limits values for P calculated according to Currie [23] are in the order of 40 mg/kg and for Si the detection limits varied from 0.04 to 0.70% depending on the sample. Samples B7–B9 collected in clean region presented low mass fractions of Si and of other elements and their gamma spectra presented low background that resulted low detection limit values.

For quality control of the Al results, several CRMs were analyzed and the apparent or non corrected and corrected

Table 1 Correction factors for P (F_{P}) and for Si (F_{Si}) for the Al determination

F_{P}	F_{Si}	Neutron flux (n cm^{-2} s^{-1})			References
		Ø$_{\mathrm{th}}$	Ø$_{\mathrm{ep}}$	Ø$_{\mathrm{fast}}$	
2.38 ± 0.03	5.29 ± 0.03	1.9×10^{12}	5.4×10^{10}	3.7×10^{11}	This study
1.576 ± 0.157	5.403 ± 0.538	$(1.05 \pm 0.0247)10^{12}$	5.04×10^{10}	2.40×10^{11}	[20]
2.55 ± 0.13	7.21 ± 0.50	4.6×10^{11}	1.4×10^{11}	NP	[21]
1.0	3.1	NP	NP	NP	[7]
0.8	1.8	8×10^{13}	2.4×10^{12}	NP	[8]

Results in mg Al/g interfering element

Ø$_{\mathrm{th}}$, Ø$_{\mathrm{ep}}$ and Ø$_{\mathrm{fast}}$ are thermal, epithermal and fast neutron flux, respectively. *NP* not presented

Table 2 Mass fractions of P and Si obtained in the analyses of certified reference materials

Certified reference material	M ± SD(n)	Z_{score}	Certified values
Results (in mg/kg) for P determinations			
NIST 1566b Oyster Tissue	6039 ± 789(4)	–[a]	
NIST 1573a Tomato Leaves	2617 ± 324(4)	1.41	2160 ± 40
NIST 1570a Spinach Leaves	5443 ± 319(5)	0.81	5180 ± 110
Results (in %) for Si determinations			
NIST SRM 1633b Coal Fly Ash	22.17 ± 0.82(4)	−1.0	23.02 ± 0.08
CTA-FFA-1 Fine Fly Ash	22.53 ± 0.22(4)	0.09	22.48 ± 0.92
INCT MPH-2 Mixed Polish Herbs	2.86 ± 0.21(4)	–	

M ± SD(n) mean and standard deviation of *n* determinations

[a] Indicates that the Z_{score} value was not evaluated since there is no certified value

Table 3 Mass fractions and detection limit values of P and Si obtained in the analyses of tree bark samples

Tree bark samples	Phosphorus (mg/kg)		Silicon (%)	
	Mass fraction	Detection limit	Mass fraction	Detection limit
B1	1946 ± 157	48	4.25 ± 0.20	0.70
B2	1547 ± 72	41	5.29 ± 0.23	0.62
B3	1454 ± 72	45	4.33 ± 0.08	0.63
B4	1720 ± 79	30	1.10 ± 0.06	0.21
B5	1364 ± 173	42	1.61 ± 0.06	0.25
B6	458 ± 9	30	0.12 ± 0.02	0.043
B7	283 ± 23	34	0.20 ± 0.03	0.105
B8	336 ± 31	32	0.107 ± 0.005	0.064
B9	324 ± 25	35	0.124 ± 0.002	0.063

Samples B1–B3 collected in São Paulo city from *Ligustrum vulgare* tree species; B4–B6 collected in São Paulo city from *Tipuana tipu* species and B7–B9 in the interior of the State of São Paulo from *Poincianella pluviosa* species

Al mass fractions obtained are presented in Table 4. The corrected mass fractions of this Table indicate a good accuracy with |Z-score| ≤ 1 but are all marginally low. This could be due to high activities of ^{38}Cl, ^{56}Mn and ^{24}Na that caused increased dead time in the measurements of ^{28}Al activities. Further studies on this matter could be undertaken to eliminate these interferences.

As can be seen in Table 4, the corrections improved the accuracy of the results for NIST 1646a Estuarine Sediment and INCT MPH-2 Mixed Polish Herbs CRMs. The interference contribution for Estuarine Sediment CRM was due to high mass fraction of Si (40.00 ± 0.16)% in comparison to Al (2.297 ± 0.018)%. In the case of Mixed Polish Herbs also the high level of Si of (2.86 ± 0.21)% in relation to that of Al (670 ± 111) mg/kg caused interference resulting in high mass fractions for Al.

The results obtained for Al in tree bark samples presented in Table 5 show, in general, good precision with

Table 4 Mass fractions of Al determined in the certified reference materials together with its certified values

Certified reference material	Non corrected Al mass fraction				Corrected Al mass fraction				Certified values
	M ± SD	RSD (%)	RE (%)	Z_{score}	*M* ± SD	RSD (%)	RE (%)	Z_{score}	
NIST 1633b Coal Fly Ash	14.84 ± 0.73*	4.9	1.4	0.28	14.71 ± 0.74*	5.0	2.3	−0.45	15.05 ± 0.27*
NIST 1646a Estuarine Sediment	2.476 ± 0.038*	1.5	7.8	4.58	2.266 ± 0.041*	1.8	1.4	−0.74	2.297 ± 0.018*
CTA-FFA-1 Fine Fly Ash	14.99 ± 0.22*	1.5	0.8	0.41	14.86 ± 0.21*	1.4	0.07	−0.035	14.87 ± 0.39*
INCT MPH-2 Mixed Polish Herbs	798 ± 53	6.6	19.1	1.67	624 ± 53	8.5	6.9	−0.60	670 ± 111
NIST 1515 Apple Leaves	275 ± 21	7.6	3.8	−0.51	271 ± 20	7.4	5.2	−0.73	286 ± 9
NIST 1577b Oyster Tissue	212 ± 9	4.2	7.5	1.56	191.8 ± 8.9	4.6	2.7	−0.57	197.2 ± 6.0
INCT-TL-1 Tea Leaves	2163 ± 126	5.8	5.5	−0.67	2158 ± 126	5.8	5.8	−0.70	2290 ± 280
NIST 1573a Tomato Leaves	582 ± 27	4.6	2.7	−0.58	578 ± 27	4.7	3.3	−0.72	598 ± 12
NIST 1570a Spinach Leaves	296 ± 27	9.1	4.5	−0.51	281 ± 26	9.3	9.4	−1.1	310 ± 11

Results in mg/kg unless indicated. Numbers with an asterisks (*) are results in percentage

M ± *SD* arithmetic mean and standard deviation from 4 to 6 determinations, *RSD* relative standard deviation, *RE* relative error

Table 5 Mass fractions and detection limits of Al (in mg/kg) obtained in the analyses of tree bark samples, data of the ratio between mass fraction of interfering element and of Al and contribution rate (CR)values

Tree bark samples	Non corrected mass fraction	Corrected mass fraction		[P]/[Al]	[Si]/[Al]	CR (%)	Detection limit
	M ± SD(*n*)	*M* ± SD(*n*)	RSD (%)				
B1	14,385 ± 600(6)	14,135 ± 600(6)	4.2	0.14	3.01	1.8	37.6
B2	16,092 ± 860(6)	15,783 ± 860(6)	5.4	0.10	3.35	2.0	41.6
B3	14,970 ± 925(6)	14,717 ± 925(6)	6.3	0.10	2.94	1.7	34.9
B4	3654 ± 196(4)	3585 ± 196(4)	5.5	0.48	3.06	1.9	12.2
B5	5099 ± 501(4)	5001 ± 500(4)	10.0	0.27	3.22	2.0	23.0
B6	376 ± 38(6)	369 ± 38(6)	10.3	1.24	3.33	1.9	9.4
B7	537 ± 40(4)	526 ± 46(4)	8.7	0.54	3.88	2.1	11.1
B8	258 ± 12(4)	253 ± 13(4)	5.1	1.33	4.24	2.0	11.4
B9	360 ± 15(4)	353 ± 15(4)	4.2	0.92	3.52	2.0	11.5

Samples B1–B3 collected in São Paulo city from *Ligustrum vulgare* tree species; B4–B6 collected in São Paulo city from *Tipuana tipu* species and B7–B9 in the interior of the State of São Paulo from *Poincianella pluviosa* species

M ± *SD(n)* arithmetic mean and standard deviation of *n* determinations, *RSD* relative standard deviation, *[P]/[Al]* ratio between mass fractions of P and of Al, *[Si]/[Al]* ratio between mass fractions of Si and of Al, *CR* contribution ratio

relative standard deviations lower than 10.5%. Contribution rate values that indicate the degrees of the interference were of the order of 2.0%. Aluminum detection limits for tree bark samples analyses were lower than their contents in the samples indicating the feasibility of applying the proposed procedure for Al determination.

Besides Table 5 shows that bark samples B1–B3 from *Ligustrum vulgare* species presented higher Al mass fractions than those obtained in barks from *Tipuna tipu* (samples B4–B6). Since these samples were collected in the same city, this difference can be attributed to the bark structure porosity that may be affecting the accumulation of pollutants. Aluminum mass fractions in tree barks are different among tree species [24, 25]. Results obtained for the samples B7–B9 presented lowest Al concentrations since these samples were collected in regions considered with low levels of pollutants and besides these samples were from *Poincianella pluviosa* trees that present a squamous bark, which is not porous.

Concerning Al and Si found in the tree barks, their origins may be associated with the suspension of soil dust deposited by the wind and with vehicular emissions and with tailpipe emissions and brake and tire wear [26, 27].

Conclusions

From the results obtained in this study, it can be concluded that NAA can be applied in the Al determination in tree bark samples. The detection limit values for Al were lower than the mass fractions of this element in the barks and the relative standard deviations of the results were, in general lower than 10.0%. However, NAA in Al determinations should be used with caution. The contributions from P and Si nuclear interference reactions should be evaluated since they depend on the relations between the mass fractions of the interfering elements and of Al. Besides, in the case of environmental samples the mass fractions of these elements depend on the local pollution where the samples were collected.

The contributions due to the nuclear reactions of P and Si in the Al determination in tree barks can be obtained by means of the P and Si interference correction factors and their mass fractions in the samples by NAA. The procedure presented in this study can be applied to determine Al present in tree bark matrix in environmental pollution studies

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