PIXE ANALYSIS OF TRACE ELEMENTS IN GENUS HIPPOPHAE L*

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ABSTRACT

Trace elements in four kinds of the *Genus Hippophae L* pulps were analysed by PIXE. Optimization of the system performance was done by carefully selecting the absorbers. Analytical sensitivities of the PIXE system were obtained by means of "external standard method".

Keywords: PIXE Trace elements Genus Hippophae L

1 INTRODUCTION

In recent years, the PIXE analysis has been widely applied to many materials of biology and medicine. Why is *Genus Hippophae L.* (GHL) analysed with PIXE. Firstly, in the past a few years a great many active substances of living things have been discovered inside the fruits, roots and leaves of GHL. Secondly, the analysed GHL samples were mostly their juice and oil. Finally, as for an analysis method, the PIXE analysis of GHL was never reported. Up to now, 13 trace element contents (μg/g) in four kinds of GHL fruits all growing in Gansu Province are analysed successfully.

2 EXPERIMENTS

2.1 Setup

The analysis was performed on the 1.7 MV tandem accelerator at Lanzhou University. The Si (Li) X-ray spectrometer based on MCB and IBM-PC/XT was used for datum acquisition and handling. Off-line analysis of the spectra was done with a program compiled at the University.

2.2 Optimization of the PIXE system

A good PIXE system produces sharp peaks of characteristic X-rays on desirable backgrounds. According to the optimization of the PIXE system done previously[1], it is obvious that the proton of higher energy and thicker absorber are benificial to the bio-PIXE analysis of the medium and heavy elements. As the qualitative analysis of

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GHL pulp is known, there are plenty of light elements inside the samples. In order to measure much more medium and heavy elements in the samples of GHL pulp meanwhile considering the analysis of the main light elements we also carried on the condition experiments on the samples of GHL pulp. In Fig.1a the characteristic peaks of some light elements such as Si, S, Cl, K and Ca can be seen clearly. But most of the peaks among medium and heavy elements are immersed under a high background except for the elements Fe and Mn with slightly higher contents in

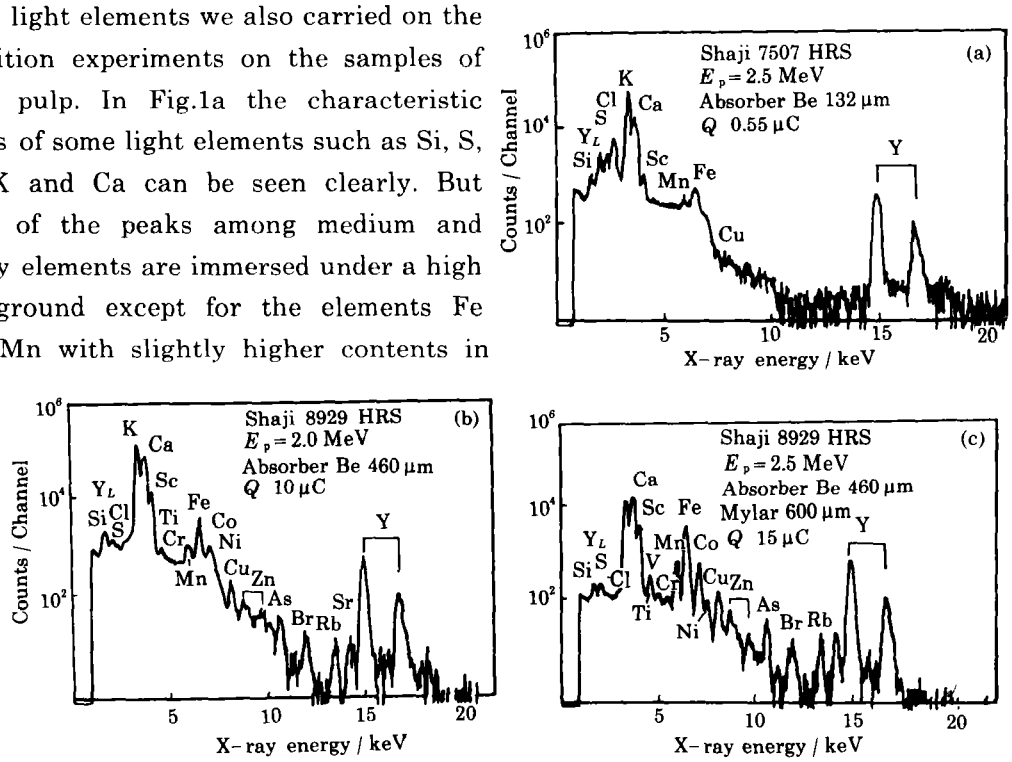

Fig.1 Measured spectra for GHL (HRS) with different absorbers

the samples. This background mostly comes from the contribution caused by the bremsstrahllung radiation of sample matrix and also by the pile-up of light elements under the condition of high counting rates of electronics. Although the quality of the PIXE spectrum in Fig.1b was partly improved yet many other elements at the energy region between elements Ti and Ni were still not clear enough. If an extra 600 μm thick Mylar film was added to the original absorber of Fig.1b, one satisfied PIXE spectrum 1c was obtained. It proves that one absorber with sufficient thickness inserted between the sample and Si (Li) detector can effectively reduce the influences of matrix and light elements in the samples of living things on the PIXE analysis of medium and heavy elements. For the thin target with the Mylar backing of 3.5 μm thickness with hundreds of $\mu g/cm^2$ when proton beam current density is over 30 nA, the target is very easy to be deformed and even to be broken down because of the energy deposit of proton in it. Through the experiment and comparason many a time, the following main parameters are at last used to analyse GHL pulp: proton beam energy is 2.0 MeV, beam current density 10—20 nA, absorber 443 μm Be + 600 μm

Mylar. By the way, all the parameters mentioned above are also suitable to the analysis of the medical samples such as the hair and the serum of human being.

2.3 Quantitative analysis of GHL pulp

During the quantitative analysis, the single standard method in relative method[2]

Table 1

PIXE analysis results of 9 elements for SRM of NBS

Element	SRM 1577a(bovine liver)		SRM 1572 (citrus leaves)	
	NBS	PIXE exp. ($n=5$)	NBS	PIXE exp.($n=5$)
K / %	0.996±0.007	0.98±0.1	1.82±0.1	1.9±0.2
Ca	120.0±7.0 μg / g	121.0±4.3 μg / g	3.15±0.1 %	3.2±0.3 %
Mn / μg · g^{-1}	9.9±0.8	9.6±3.5	23.0±2.0	23.3±3.8
Fe / μg · g^{-1}	194.0±20.0	197.5±16.0	90.0±10.0	86.5±7.8
Cu / μg · g^{-1}	158.0±7.0	158.5±4.4	16.5±1.0	16.5±1.4
Zn / μg · g^{-1}	123.0±8.0	122.7±9.3	29.0±2.0	28.5±2.2
Rb / μg · g^{-1}			4.84±0.1	4.6±1.3
Sr / μg · g^{-1}			100.0±2.0	99.2±13.8
Pb / μg · g^{-1}			13.3±2.4	12.9±3.0

is principally adopted while the relative sensitivity factors are measured, so called "external standard method" was used in which Y was not put into each standard sample, instead, only was made into an individual standard sample. After measuring the absolute sensitivity factors of each element N_j / W_j, these factors will be normalised by the absolute sensitivity factor of the element Y so that the relative sensitivity factors of element J, $S_J = (N_j / W_j) / (N_Y / W_Y)$, could be obtained, where N_j and N_Y stand for the area of net peak of element J and Y respectively while W_j and W_Y for the weight of J and Y each. Do remember this, while preparing the targets it is necessary to put the internal standard Y into GHL samples. Fig.2 shows the relative sensitivity curve made up of 22 elements. It can be seen that the experimental data are very close to the fitting ones on the curve. In order to check the accuracy of this analysis, we once analysed parts of elements of SRM, NBS 1577a and 1572 by PIXE, the results (see Table 1) show that the analysed data quite agree with the certified ones within the range of deviation.

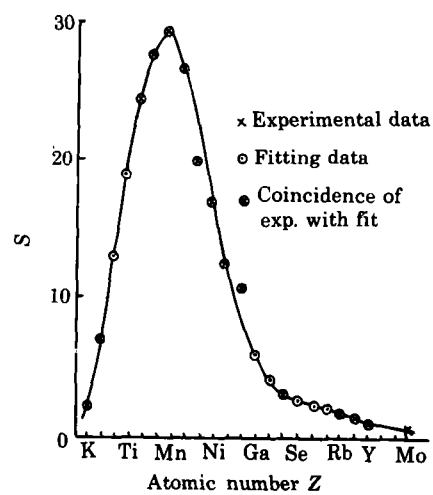

Fig.2 Relative sensitivity curve of K_α — lines

2.4 Samples and preparation of target

So far, only four kinds of the GHL are found in Gansu Province and their scientific names are *H. rhamnoides L. subsp Sinensis Rousi* (HRS), *H. rhamnoides L. subsp Turkestanica Rousi* (HRT), *H. Thibetana Schlebhtend* (HTS) and *H. neurocarpa* (HNL) respectively[3]. For target preparation, the GHL fruits were allowed to dry and the seeds taken away from the fruits (except for HNL), were weighed and put into an oven at 60—80 ℃ for 24 h, and then moved into a low temperature plasma of ashing for 8 h. The ashes were dissolved in 6 molar MOS nitric acid and $Y(NO_3)_3$ was added. The solution of 100 μl was moved onto a Mylar film that had been treated by 20 % NaOH and 1 % PVP solution. The thicknesses of the targets are about 200—300 $\mu g/cm^2$, the diameter about 5 mm.

3 RESULTS AND DISCUSSION

Table 2 shows that each of GHL pulps contains rich trace elements such as

Table 2

PIXE analysis results for 4 kinds of GHL pulps μg/g

	HRS ($n=4$)[1]		HTS ($n=2$)		HRT ($n=4$)		HNL ($n=2$)		HRS ($n=4$)[1]		HTS ($n=2$)		HRT ($n=4$)		HNL ($n=2$)	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
K	11660	±1575	5855	±343	6276	±412	1497	±1.2	Ni	4.6	±1.6	8.3	±3.7	2.4	±0.3	1.8	±0.0
Ca	2048	±275	860	±8.3	837	±68	756	±1.2	Cu	29.6	±10.1	21.3	±8.7	13.2	±3.5	3.2	±0.0
Ti	41.4	±14.6	31.4	±9.7	19.8	±7.3	7.7	±0.2	Zn	15.7	±1.6	4.5	±0.0	7.0	±0.9	2.7	±0.1
V	6.7	±1.6	4.3	±0.7	3.3	±1.2	1.1	±0.1	As	16.3	±1.4	13.9	±0.3	3.1	±0.3	1.4	±0.0
Cr	1.5	±0.1	0.9	±0.6	0.8	±0.2	0.2	±0.0	Rb	17.9	±2.8	1.2	±0.5	3.1	±0.9	0.3	±0.0
Mn	21.2	±2.3	11.5	±1.2	7.2	±1.2	8.6	±0.2	Sr	8.6	±2.1	4.4	±0.9	4.5	±0.6	2.2	±0.0
Fe	197.1	±64.1	234.9	±33.3	76.2	±27.9	62.3	±0.2									

Fe, Cu, Mn, Zn, *etc.*, being benificial to man health. Meanwhile K and Ca have much more contents. Besides, the different GHL fruits have a great difference in the contents of trace elements. Finally, it is necessary to point out that the optimization experiments of PIXE system and condition experiments of the samples to be analysed are most important.

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