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An Investigation About the Factors Influence on the Wavelength Dispersive X-Ray Fluorescence Spectrometer Measurement Results*

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ABSTRACT: In this study, in the product waste after treatment of boron the different masses, cellulose ratios and different scanning surface areas effect on analysis results and the effect of press pressing on the analysis results in the potassium nitrate (KNO3) samples were investigated. The aim of our work is determined the different masses, cellulose ratios, press pressing, and different scanning surface areas effect on the analysis results by using the Wavelength Differential X-Ray Fluorescence Spectrometer (WDXRFS).

Keywords: Analysis, boron**,** wavelength dispersive x-ray fluorescence spectrometer.

Dalgaboyu Ayrımlı X-Işını Floresans Spektrometre Ölçüm Sonuçları Üzerine Etki Eden Faktörler Hakkında Bir İnceleme

ÖZET: Bu çalışmada borun işlenmesinden sonraki ürün atığında, farklı kütleler, selüloz oranları ve farklı tarama yüzey alanlarının analiz sonuçları üzerine etkisi ve potasyum nitrat (KNO_3) numunesinde pres basıncının analiz sonuçlarına etkisi incelenmiştir. Çalışmamamızın amacı farklı kütleler, selüloz oranları, pres basıncı ve farklı tarama yüzey alanlarının analiz sonuçları üzerine etkisini Dalgaboyu Ayrımlı X-Işını Floresans Spektrometre (WDXRFS) kullanarak belirlemektir.

Anahtar Kelimeler: Analiz, bor, dalgaboyu ayrımlı x-ışını floresans spektrometre.

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INTRODUCTION

Investigation of severities of X-ray lines for different elements is an important task in atomic, molecular and radiation physics, geological and medical research, the qualitative and quantitative analysis in other fields. So, It is necessary the accurate measurements of these lines. Factors that influence measurements such as the sample preparation process, the effect of the counting system and environmental conditions are important. The sample preparation process is an important step in the chemical analysis process. The sample preparation process is an important step in the chemical analysis process. Analysis of atomic spectroscopic procedures practically always necessitates a simple or more complex preparation process of the samples. This step is the most critical part of the analysis due to an error in this step affects all measurement results (Hoening and Kersabiac, 1996; Cal-Prieta et al., 2002; Oliveira, 2003). However, if one examines the literature one often finds that details on how a sample was prepared are often omitted for most analytical methods of analysis (Buhrke, 1998).

In the literature, a variety of experimental data relevant to the qualitative and quantitative analysis of different samples available. Multi-element analyses of plants have been studied by using WDXRFS. However, the multi-element composition of plant samples has been analyzed by using x-ray fluorescence spectrometry (Garivait et al., 1997). A simple and cheap procedure has been described for simultaneous determination of V, Cr, Mn, Fe, Co, Ni, Cu and Zn contents in water by means of WDXRFS after preconcentration (Yamini et al., 2009). A comparative study on the elemental composition of various hazelnut (Corylus avellana L.) samples has been conducted by using a sensitive procedure, WDXRF (Akbaba et al. 2011). A method has been developed for quantitative measurement of the elemental composition of particulate matter (PM) in seawater. This procedure is based on the use of WDXRF analyzing PM harvested on various filter types (Paulino et al., 2013). Concentrations of trace mineral nutrients such as Cu, Fe, and Zn have been analyzed by WDXRF in eight different infant milk powders (Fernandes et al., 2015). Elemental analysis of different soil and geological samples have been determined by using WDXRF (Kubala et al., 2015). WDXRF has been used for quantitative and qualitative analysis of the elemental composition of cosmetic products (McWilliams et al., 2015). Enamel and dentine in human, bovine, porcine and ovine teeth have been determined by using WDXRF. The chemical compositions of these samples have been compared by using WDXRF technique (Teruel et al., 2015). The rare earth elements in combustion ashes from selected Polish coal mines have been determined by using WDXRFS (Smolinski et al., 2016). It is seen that there are few studies on the factors affecting the WDXRF measurement results in the literature review. The goal of this work completes this deficiency of the literature and other studies create basis.

MATERIAL AND METHOD

Preparation of sample

Firstly, samples milled to ensure homogeneity about 10 min. After, these samples are mixed at 2 min by SPEX mixer. The powder samples were compressed using 7-10 tons pressure manual hydraulic press. The pellets were prepared for product waste after treatment of boron (100, 150, 200, 250, 300, and 350 mg mass) and KNO_3 (150, 200, 250, 300, 350, 400, and 450 mg mass). However, product waste after treatment of boron (200 mg mass) was added different ratios of cellulose.

Wavelength dispersive x-ray fluorescence spectrometer

The element concentrations were detected by using WDXRFS of ZSX 1000 of Rigaku firm. Elements, in a wide range, from Be to U, can be analyzed in a variety of sample types and in the concentration range from 100% down to the sub-ppm-level. This system has the ability to analyze the energy range 0.1-5.9 keV. Additionally, it is the counting time 10-4000 s, counting rate $5x10^3 s^{-1}$ and the detection limits in 1000 second around (ng, mg, g^{-1}). The schematically arrangement of the WDXRFS used in this study is shown in Figure 1.

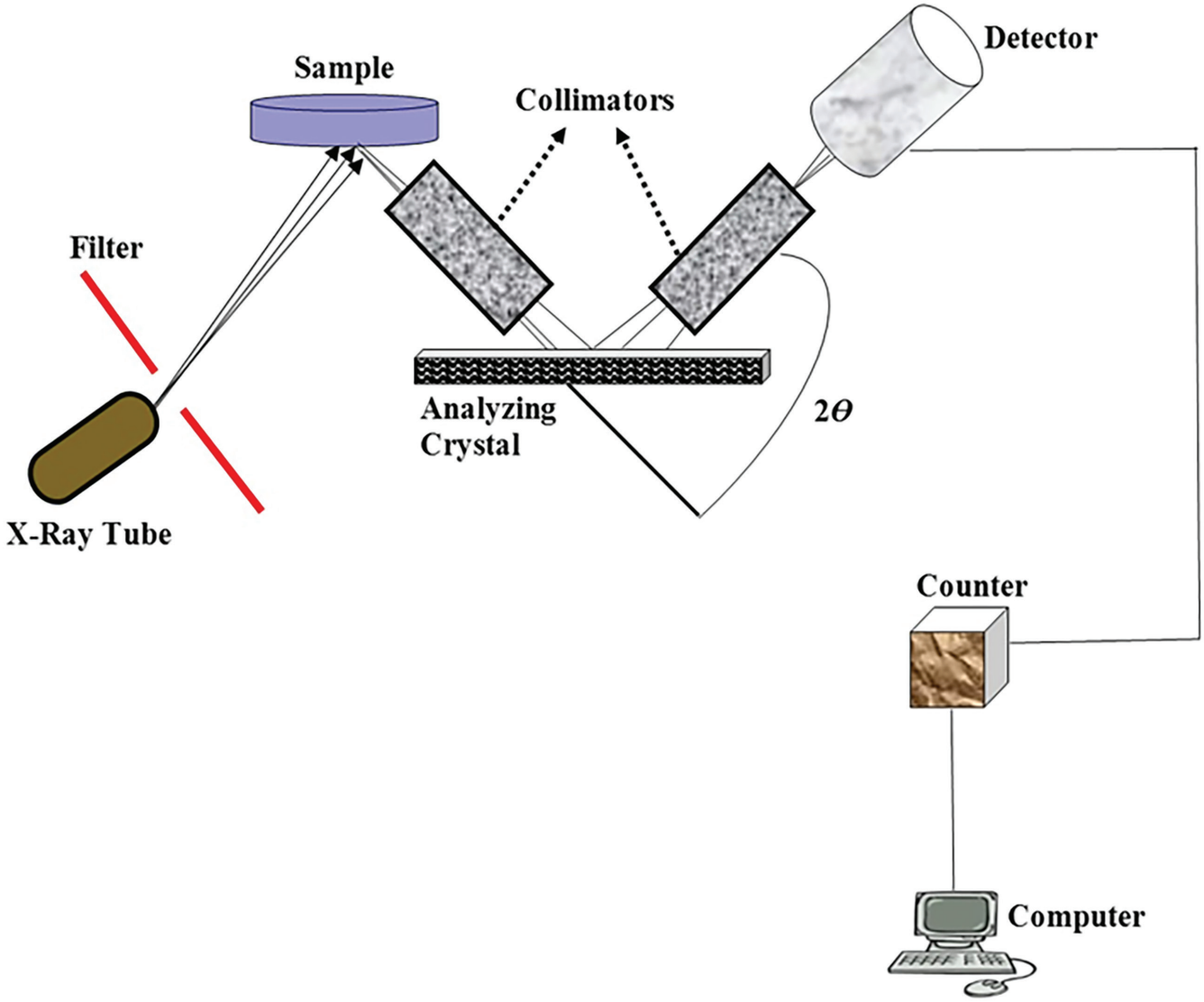


Figure 1. The schematically arrangement of the WDXRFS

RESULTS AND DISCUSSION

In present work, we examined the factors such as pressure, mass, the ratio cellulose, scanning surface areas and press pressing for two samples that influence on element concentration and measurement results with WDXRFS. Results of measurement are given with tables. Firstly, the effect of some factors were examined for product waste after treatment of boron. The effect on elements concentrations of cellulose ratio (0%, 1%, 2%, and 4%) are given Table 1 for product waste after treatment of boron (200 mg mass).

Table 1. The concentration of elements (μg/cm^2) for product waste after treatment of boron (200 mg)

	0%	1%	2%	4%
Elements		concentration of elements		
C	-	-	-	27.1034
O	-	-	-	54.4528
Na	0.2413	0.2018	0.2382	0.0593
Mg	4.3217	4.2056	4.3739	1.0925
Al	22.0294	22.0998	22.1337	5.1824
Si	55.6585	56.0096	55.6860	10.2001
P	0.2630	0.2530	0.2570	0.0362
S	0.6809	0.5723	0.5810	0.0733
Cl	0.0236	0.0453	-	-
K	3.7291	3.6841	3.6062	0.4459
Ca	3.9753	3.8197	3.8583	0.4626
Ti	0.7079	0.6721	0.6972	0.0795
Cr	0.0896	0.1099	0.1112	0.0110
Mn	0.1128	0.1075	0.2431	0.0090
Fe	7.9132	7.9258	7.9823	0.7688
Ni	0.1838	0.1696	0.1636	0.0149
Cu	-	0.0114	-	0.0011
Zn	0.0180	0.0154	0.0109	0.0014
As	0.0122	-	0.0114	0.0010
Rb	0.0098	0.0078	0.0084	0.0008
Sr	0.0274	0.0245	0.0248	0.0022
Zr	0.0124	0.0138	0.0128	0.0020
Pb	-	0.0508	-	-
$C_6H_{10}O_5$	0.0205	1.0115	1.0201	1.0399

Table 1. it is clearly seen that the concentrations of elements remarkable change was not observed in 0%, 1%, and %2 cellulose ratios. Contrary to it, generally, concentrations of elements decreased for 4% cellulose ratio. However, the concentrations of elements are different in 0% cellulose ratio. This difference is not most remarkable but most important. The cellulose is enough to be able to pellet the sample. If it is obligatory to added cellulose in the sample, several measurements should be taken at different cellulose rates. The effect on elements concentration of sample mass is listed Table 2 for product waste after treatment of boron (100, 150, 200, 250, 300, and 350 mg mass).

Table 2. The concentration of elements (μg/cm^2) for product waste after treatment of boron

	100 mg	150 mg	200 mg	250 mg	300 mg	350 mg
Elements	concentration of elements					
B	6.2957	5.7523	6.7252	5.6109	5.5878	5.2678
C	7.4472	7.3033	7.9632	6.5790	6.8678	6.5742
O	51.5784	52.7228	54.9034	53.2170	52.3066	52.4457
Na	0.0875	0.1138	0.1432	0.0810	0.1749	0.1060
Mg	3.5934	3.6945	3.4375	3.6489	3.7002	3.9557
Al	2.2990	2.0833	1.8632	1.9738	2.0312	2.1246
Si	7.8315	7.8214	7.1094	7.9187	7.9647	8.2221
P	0.0360	0.0348	0.0333	0.0370	0.0352	0.0355
S	0.3412	0.3395	0.3079	0.3413	0.4364	0.3489
K	0.8101	0.8059	0.7115	0.8163	0.8439	0.8256
Ca	18.8992	18.7513	16.1401	18.9541	19.2106	19.2513
Ti	0.0755	0.0774	0.0532	0.0514	0.0533	0.0599
Mn	0.0257	0.0151	0.0174	0.0274	0.0249	0.0230
Fe	0.5459	0.3363	0.0455	0.5774	0.6033	0.6091
Ni	0.0049	0.0072	-	0.0063	0.0054	-
Zn	-	-	0.0144	-	-	-
As	0.0044	0.0058	0.0036	0.0057	0.0062	0.0043
Rb	0.0026	0.0035	0.0030	0.0033	0.0052	0.0036
Sr	0.1082	0.1187	0.1027	0.1339	0.0032	0.1427
Pd	0.0135	-	0.0124	0.0167	0.1393	-

As seen in Table 2, in the concentrations of elements remarkable change was not observed for product waste after treatment of boron (100, 150, 200, 250, 300, and 350 mg mass). The reason is that during the WDXRFS measurements often the of the surface of the samples are scanned. Besides it was observed that the samples with 100 and 150 mg masses had surface cracking and color change during the analysis. The effect on element concentrations of scanning the surface area of the sample (3 mm, 10 mm, and mylar-coated 10 mm sample diameter) shown in Table 3 for product waste after treatment of boron (100 mg mass).

In this section, 13 mm diameter tablet samples were prepared and 3 mm and 10 mm diameter scanning surface were selected from the measuring system for these samples.

Table 3. The concentration of elements (μg/cm^2) for different scanning the surface area of product waste after boron treatment

	3 mm diameter	10 mm diameter	10 mm diameter (mylar-coated)
Elements		concentration of elements	
B	6.8301	7.9688	-
C	6.5684	8.2438	58.3642
O	54.1133	52.7855	30.6848
Na	-	0.0993	-
Mg	3.3018	3.5506	0.3405
Al	1.8532	1.9959	0.3846
Si	7.4678	7.3057	1.9278
P	0.0256	0.0314	0.0115
S	0.2875	0.2931	0.1033
K	0.7709	0.7224	0.3250
Ca	17.9751	16.3551	7.5675
Ti	-	0.0620	0.0191
Mn	0.0269	0.0172	0.0072
Fe	0.6503	0.4677	0.2157
Ni	0.0081	0.0056	0.0018
As	0.0064	0.0033	0.0011
Sr	0.1044	0.0843	0.0413
Pd	0.0102	0.0085	0.0048

It is observed from Table 3, generally, that concentrations of element decreased to 10 mm diameter (mylar-coated). The concentrations of elements remarkable change weren't observed for 3 mm and 10 mm diameter.

Secondly, the effect of press pressure is examined for KNO_3. The effect on element concentrations of 7-ton press pressure and 10-ton press pressure is listed Table 4 and 5 for KNO_3 (13 mm diameter, 100, 150, 200, 250, 300, 350, 400, and 450 mg mass).

Table 4. The concentration of elements (μg/cm^2) for 7-ton press pressure applied to KNO_3

	150 mg	200 mg	250 mg	300 mg	350 mg	400 mg	450 mg
Elements			concentration of elements				
B	-	-	-	-	1.7866	-	-
N	11.7812	10.7023	10.9700	12.9230	11.1117	9.9762	-
O	41.5156	42.0656	42.3220	42.1239	42.3418	42.6402	42.5300
Na	0.1512	0.1437	0.1229	0.0784	0.0631	0.1156	0.1085
Si	-	0.0167	-	-	-	-	-
P	-	-	0.0020	-	-	-	-
S	-	0.0046	-	-	-	-	-
K	46.5319	47.0670	46.5607	44.8533	44.6968	47.2174	57.3274
Pd	0.0200	-	0.0223	0.0214	-	0.0284	0.0341

Table 5. The concentration of elements (μg/cm²) for 10-ton press pressure applied to KNO_3

	150 mg	200 mg	250 mg	300 mg	350 mg	400 mg	450 mg
Elements	concentration of elements						
B	-	-	-	-	0.7396	-	-
N	-	8.1262	8.0281	-	6.6912	-	7.0903
O	27.9042	27.7428	28.8248	30.4128	28.7657	29.0299	27.6109
Na	0.1179	0.1120	0.1199	0.0709	0.0757	0.1218	0.1580
Mg	0.0232	0.0174	-	0.0175	-	-	-
Al	0.0101	-	0.0081	-	-	-	-
Si	0.0301	0.0190	0.0230	0.0189	-	-	-
S	0.0044	0.0050	0.0030	-	-	-	0.0027
Cl	-	0.0205	-	0.0181	-	-	-
K	71.9101	63.9572	61.9222	68.1632	62.6672	69.3746	64.1925
Cu	-	-	-	-	-	0.0362	-
Pd	-	-	1.0709	1.2691	1.0616	1.4375	0.9457

In Tables 4. and 5, the concentrations of element significant change was observed. The elemental concentrations have a relative change of 7 and 10 tons' pressure. Also, when pressure increases from 7 to 10 tons, generally, the $Z > 11$ increased concentrations of elements and $Z \leq 11$ decreased concentrations of elements. These results demonstrate the importance of the applied pressure and sample thickness when preparing the sample.

CONCLUSIONS

As a result, the sample preparation process is an important step in qualitatively and quantitatively analyzed. The experiments and measurements depend mainly upon the mass of the sample, applied pressure, the ratio of cellulose, the effect of scanning the surface area of the sample, instrument operation conditions and the environmental conditions. So, these conditions should be taken into account in order to good experimental results.

These conditions should be taken into account for future experimental studies. In the future, this work repeatable for EDXRF (Energy Dispersive X-Ray Fluorescence) or different measurement systems, different samples, different energies, and different experimental factors.

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