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SEMI-PARAMETRICAL NAA METHOD FOR PAPER ANALYSIS

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ABSTRACT

The semi-parametric Neutron Activation Analysis technique, using Au as flux monitor, was applied to determine element concentrations in white paper, usually commercialized, aiming to check the quality control of its production in industrial process.

1. INTRODUCTION

Accurate determination of the elements present in white paper, usually commercialized, it is very important because the mechanical and physicochemical properties of the final product depend on its composition. Considering that the industrial process of white paper involves different steps, the projected characteristic of its composition can be altered until to obtain the final product. Nowadays, there are lot of procedures/methods used for checking these steps; a summary about these analyses is presented in references [1] and [2].

An usual way to perform the quality control of this industrial process of paper production is the analysis of the ashes; this is a global but a destructive method.

Based on it, in this study, we intend to perform a quali-quantitative determination of the elements present in white paper by means of the semi-parametric neutron activation technique followed by the gamma spectroscopy measurements of the photons generated by beta decay

and/or the electron capture of the radioactive products generated, aiming to propose an alternative procedure to performed these analyses without the need to use the ashes.

Considering that calcium salts and chlorine compounds are used in white paper production (≥ 1% by weigh) it is predictable the strong presence of Cl and Ca group elements in paper samples so, it is very important to perform a quantitative determination of these elements in all its extension.

2. EXPERIMENTAL PROCEDURE

In this work a large sample of white paper (about 10m large) was extracted of the paper-mill and divided in various sheet-samples of A-4 size. One of those sheets - sample (~ 2.5cm) was selected for a preliminary analysis.

This select sample was divided in five bands of ~5 cm each (denominated P1, P2, P3, P4 and P5) and these bands were submitted to neutron activation in the IPEN nuclear reactor. In a second step, each band (P1, P2, P3, P4 and P5) was again divided into small fractions of ~1 cm size (denominated p11, p12, p13, ..., p55) and again they were submitted to neutron activation.

A total of 5 samples of paper (~0.660g each) were analyzed in first step (P1, P2, P3, P4 and P5) and 25 samples (~0.130g each) in the second one (p11, p12, p13,..., p54 and p55). We used this procedure to verify the homogeneity of the results for all elements quantified.

In order to determine the concentration of the elements in these paper samples, the Cadmium Ratio Technique was used for the measurement of thermal and epithermal flux distribution [3]. In this technique, Au foils, both bare and Cd covered, are irradiated together with the paper sample in the IEA-R1 nuclear reactor at IPEN/SP (IEA-R1, 2-4MW, pool type), for 5 minutes, allowing the simultaneous activation of these materials under the exact same irradiation conditions. Using this procedure the γ-ray activities induced in the Au foils by both the thermal and epithermal neutrons were obtained as well as the activation of paper sample. A γ - ray spectrometer system with a semiconductor detector connected to an ADCAM multichannel analyzer and to a PC computer were then used to measure the induced gamma-ray activity. All gamma spectra analyses were performed using the IDF computer code [4] and the concentration of each element was then obtained by using an in - house software [5].

3. RESULTS AND DISCUSSION

The time optimization used in this experiment to perform these analyses: irradiation time of 5 minutes; counting time of 5 minutes for each gold foil and 10 minutes for each sample and background radiation, in a thermal neutron flux ~10^{11} $n.cm^{-2}.s^{-1}$, allowed us to conclude the analysis in about two hours or less. Following this procedure, it was possible to identify and quantify Ca and Cl as well as Br, Mg, Mn and Na in all samples.

The results for the five bands (P1, P2, P3, P4 and P5) analyzed in the first step are presented in Table 1. These data represent the mean value from the two irradiations performed for each sample. For illustrative visualization in figure 1 the behavior of sodium concentration, in this step of the analysis, can be appreciated.

The results of others analyses performed in the second step (p11, p12, p13,..., p54 and p55) were not presented in tables (due the number of the tables to perform it) but, the analysis of the 5 small fractions from P4 sample (p41, p42, p43, p44 and p45) were summarized in figure 2, where the result of the P4 sample (obtained from the first step) was also included for comparison. Yet, in this figure the confidence interval, taken at ±1 and ±2 SD, were also presented to permit an evaluation of the homogeneity of the data.

Table 1. Element concentrations in white paper using the semi-parametric Neutron Activation Analysis technique

	Elements g/kg					
Paper samples (mass)	**Br**	**Ca**	**Cl**	**Mg**	**Mn**	**Na**
P1 (0.648g)	0.024±0.009	44±9	1.97±0.19	0.99±0.08	0.0036±0.007	2.00±0.11
P2 (0.664g)	0.018±0.002	59±8	2.56±0.14	1.00±0.10	0.0010±0.0002	2.25±0.17
P3 (0.683g)	0.011±0.009	56±4	2.37±0.15	1.00±0.07	0.0038±0.0002	1.75±0.11
P4 (0.665g)	0.0017±0.002	51±8	2.91±0.19	1.32±0.13	0.0048±0.0006	2.14±0.13
P5 (0.661g)	0.027±0.017	59±10	2.92±0.15	1.21±0.10	0.0037±0.002	2.66±0.15

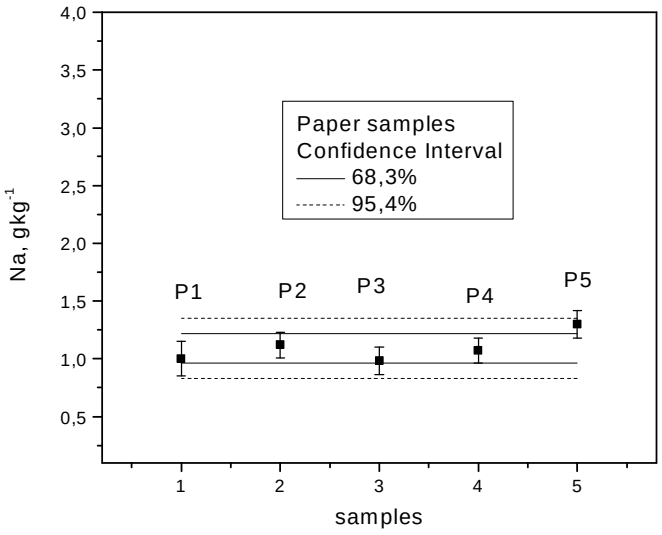

Figure 1. The concentration of sodium in paper samples (P1, P2, P3, P4 and P5).

The data have been normalized using the P1 sample value.

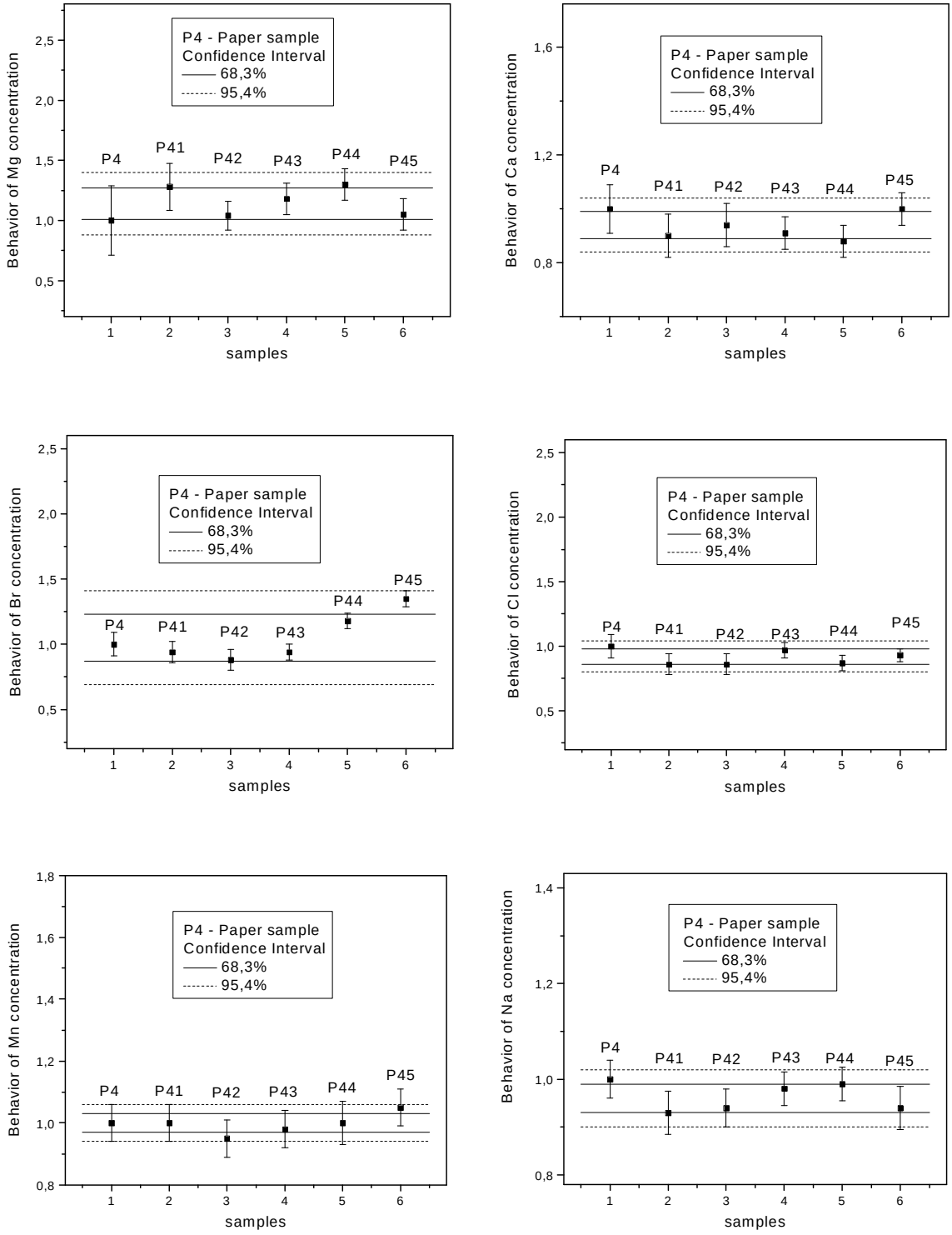

Figure 2. The behavior of the elements in the small fractions of P4 sample paper (second step). All the results were normalized using the P4 sample value.

The concentration behavior of the elements analyzed (Br, Ca, Cl, Mg, Mn and Na) in each small fraction of all samples paper (p11, p12, p13, ..., p54 and p55), are in agreement considering a confidence interval of 95% (±.2 SD).

Although this nuclear procedure needs a lot of irradiation to monitor all extension of paper (10m), in function of samples size selected (~1cm), its permits detailed information about the paper composition and consequently a very precise monitoring relate to the performance of the paper-mill.

4. CONCLUSION

The optimization of this quali - quantitative method was established for paper analysis. Now, we intend to continue this investigation for all the extension of the paper, as well as to invest in search of eventual long-range concentration gradients, such as Fe and Co, common additives used in the production of paper.

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