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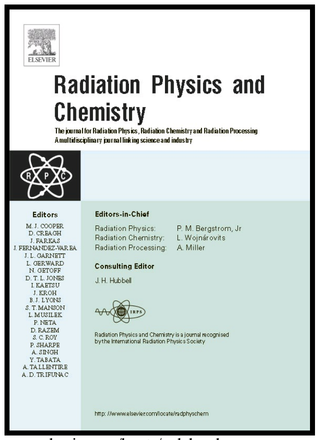

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ABOUT THE ELEMENTAL ANALYSIS OF DENTAL IMPLANTS

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3.1 Materials And Methods

As a first investigation we have performed EDS/SEM and WDS measurements. On both machines we have measured five brass samples (three small cylinders, comparable to the implant dimensions, one disk and scobs (packed in a disk shape) in order to investigate the influence of different, nonstandard geometries. We have also measured an old discarded dental implant to check the differences between the results from both equipment in a real implant.

Since dental implants are essentially a screw, its geometry is quite apart from the usual required disks (radius varying from 5mm to 50mm and thickness of maximum 47mm), then it is important to verify if the equipment would work satisfactorily with the implant geometry. If cutting or grinding is necessary, the procedure could modify the composition and/or structure of the analyzed sample,

The characteristics of the five brass samples are: (1) a disk with 40mm diameter and 7mm thickness; (2) three small cylinders, similar in dimensions to the implant (4.01 mm diameter and 11mm length) and (3) scobs (each chip having in average 2.3mm length) packed in a disk shape. The samples are depicted in Figures 1 a, b and c, respectively. The last sample is an old discarded implant, with a chemically saturated surface.

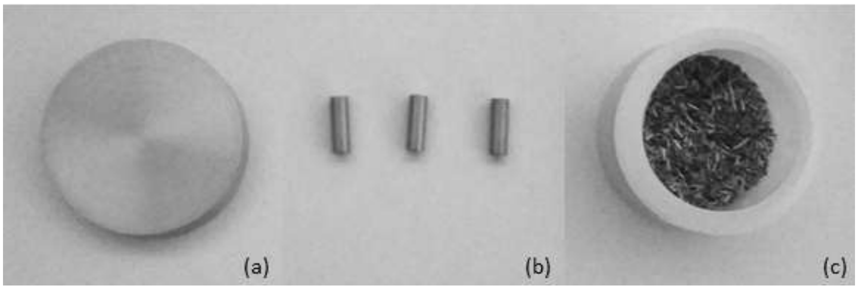

Figure 1: Brass samples with different geometries

The equipment used for WDXRF was a Bruker S8 Tiger model 1 kW (Massachusetts, USA) of the Laboratory of Photon and Electron Impact (LIFE), from the Department of Physical Chemistry of the Federal University of Rio de Janeiro

(UFRJ). The instrument allows the maximum voltage of 50 kV and 50 mA, with the power limited to 1kW. The analyzed layer can reach the depth of microns in the studied sample. To measure the cylinders and implant it was used a carbon disk as base, with the samples fixed and covered with a 3525 Ultralene film with thickness 4μm from SPEX SamplePrep (Figure2). For the analysis it was used the QUANT-EXPRESS software.

Figure 2: Sample preparation for the cylinder and implants in WDS.

All the samples were also analyzed in a SEM/EDS equipment from Hitachi, model TM3000 (Tokyo, Japan) coupled with Bruker Scan Generator and X-Flash Detector (Massachusetts, USA) of the Laboratory of Scanning Electron Microscopy from the Materials and Chemistry Technology Sector (STMQ) of the Institute of Nuclear Engineering (IEN). During the acquisition data the samples were maintained in place on the standard equipment support. All measurements were performed with the same magnification (300X). To analyze the data, the software QUANTAX 70 was used.

4- RESULTS AND DISCUSSION

The results obtained for the brass samples in both SEM/EDS and WDS are presented in the graph depicted in figure 3. The main detected elements are, as expected, copper and zinc. Lead was also detected in both methods for all brass samples, having a higher concentration for the scobs, as it can be seen in figure 4. This could be explained by the manufacturing process. The chips have more contact with the cutting tool which could be the source of lead. Carbon was detected on the SEM/EDS but not on the WDS, which is not equipped with the proper crystal to detect low Z elements. The other elements detected, completing the normalization to 100% are Na, Sn, Fe, As, Si, Ca, S, Cl, Ni, Al and O.

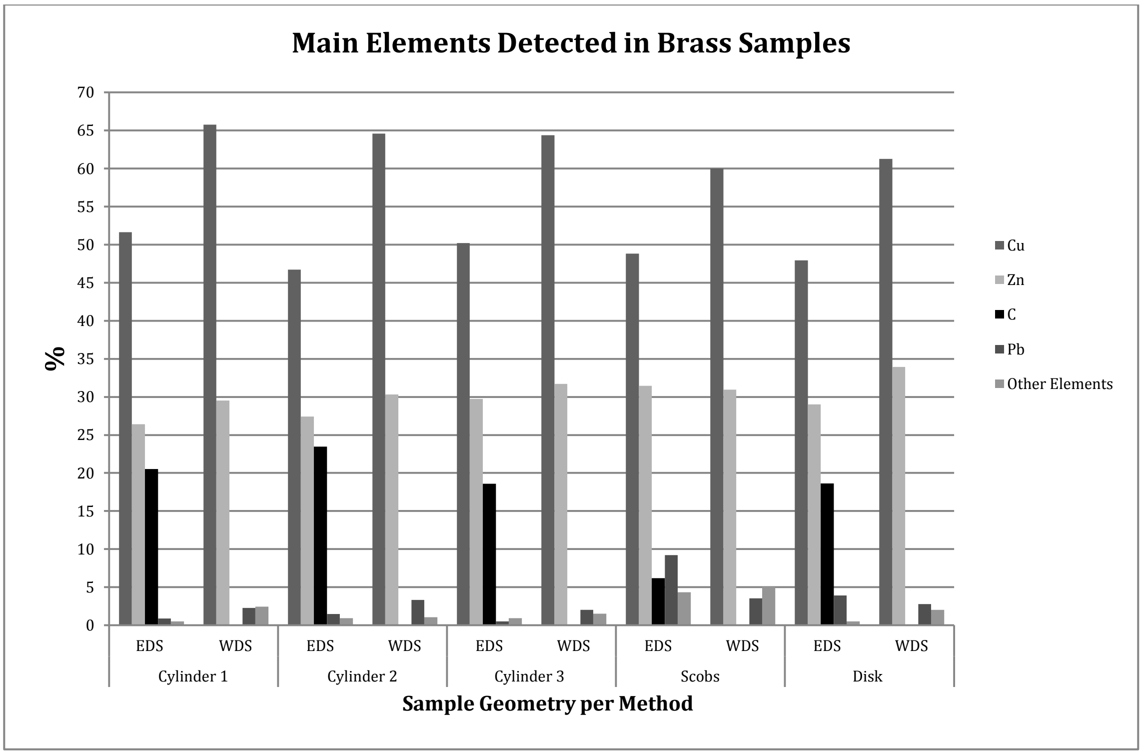

Figure 3: Main elements detected for the different brass sample geometries in both EDS and WDS.

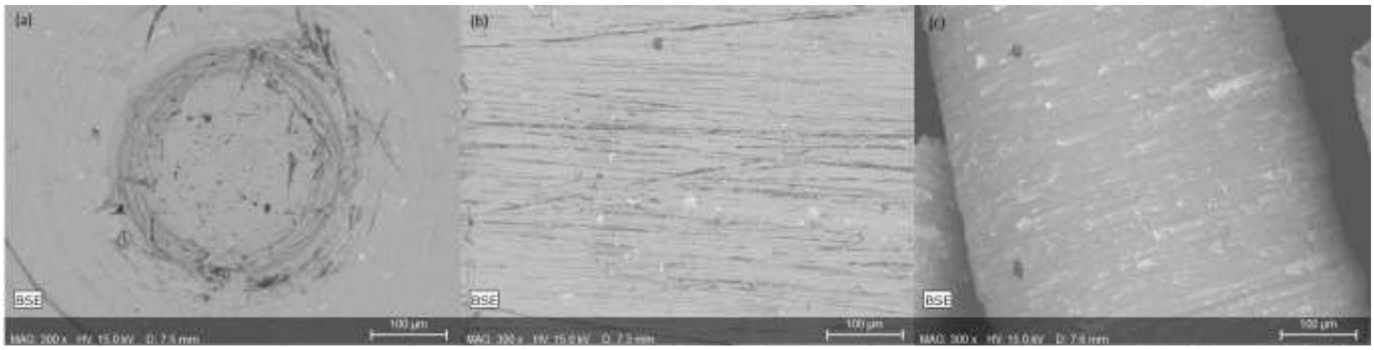

Figure 4: Comparison of the surface image of the three different sample geometries. The white spots correspond to lead: (a) disk, (b) cylinder and (c) scobs. The lead is more frequent on the scobs surface.

Copper was the element with the higher concentration found by both methods. The measures in SEM/EDS for cylinders 1, 2, and 3 were of 51.65%, 46.73% and 50.2% respectively, while the scobs presented 48.81% and the disk 47.93% with an average uncertainty of 1.5%. On the other hand, the Cu concentration obtained in the WDS were of 65.75%, 64.57%, 64.37% for the cylinders 1, 2 and 3 respectively, 60.02% for the scobs and 61.24% for the disk with an uncertainty around 0.55% (provided by the analyzing software). In both cases it would more correct to use just one decimal for the data but we decided to keep the number of digits provided by the software. The results found on the WDS for the cylinders present a closer relation to each other, but also larger than the uncertainty. The differences between these ones and the other geometries must also be due to the geometry. The results for the cylinders on the EDS show a fluctuation around 4% that could be related to the different concentration of the contaminants in each sample surface.

The different analysis show other elements (Na, Sn, Fe, As, Si, Ca, S, Cl, Ni, Al and O), in different amounts on each sample, in a very low concentration with uncertainty similar to the amount found. This should be better investigated.

Both software provide uncertainties associated with the measurement. The average uncertainty related to the measure for each element of the samples for the EDS is: 1.5% for Cu, 0.9% for Zn, 3.3% for C, 0.5% for Pb. For WDS it is: 0.55% for Cu, 0.39 for Zn, 15% for Pb.

The discrepancies between the concentrations found in both methods can be justified by the fact that the SEM/EDS data refers to a thinner layer than in the WDS, allowing superficial elements, not present in the inner core, to be detected.

Taking into consideration the characteristics mentioned above, it is possible to conclude that for both methods the validation of the cylinder geometry was satisfactory, keeping the same proportion of elemental concentration for all of the samples, but with a larger uncertainty than the statistical one provided by the used software.

After the validation of the non-conventional geometry, a discarded implant was measured with both methods. The results are presented in table 1. On the EDS results it was possible to see that the surface of the implant is composed of Ti, C and O, a well-known result already cited in many references, since TiO2 is a native layer present in all implant surfaces and C is the most found contaminant [5, 16, 17, 18]. WDS results show basically Ti, with 98.6%, together Si, S and Al in much lower concentrations. It should be noted that O and C are not detected in WDS due to limitation of the equipment.

Equipment	EDS		WDS	
Element	Weight Conc. (%)	Error (%)	Weight Conc. (%)	Error (%)
Ti	74.46	2.3	98.6	1.4
C	13.92	3.4	-	-
O	11.62	4.2	-	-
Si	-	-	0.6	22.2
S	-	-	0.4	18.4
Al	-	-	0.4	28.3

Table 1 - Comparison between EDS and WDS analysis of a discarded implant.

4. CONCLUSION

The results have demonstrated the elemental analysis, particularly of dental implants, depends on the technique, meaning the radiation and energy chosen as well as the selected region of interest on the sample. For surface layers, the most important region for dental implants, electron microscope coupled to EDS analysis is a suitable technique but requires some attention specially when evaluating the uncertainty, avoiding to just use the statistical one provided by the equipment, and verifying the reproducibility.

Using a second method as WDS, on the other hand, allows analyzing a deeper layer of the sample. But in this case special attention to the sample geometry should be paid. The combination of both methods allows a more complete evaluation of the dental implant: the surface layer, showing the oxidation and/or coating properties as well as the characteristics of the titanium core used for the implant. The next step of our study is to analyze the temporal evolution of the surface layer when exposed to the atmosphere. And then to test different brands offered in the Brazilian market.

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HIGHLIGHTS

- We investigate the influence of sample geometry on the elemental analysis of dental implant
- The history and a short review of the area is performed
- Elemental analysis of five brass samples with SEM-EDS and WDS
- The results show that the geometry has influence on the detected amount of the components
- It was found that the uncertainty was larger than the one provided by the equipment