Index

CHARACTERIZATION OF CORROSION PRODUCTS FROM Nd-Fe-B MAGNETS USED IN DENTAL PHOSTHESES

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ABSTRACT

This work presents results obtained in the elemental analysis of Nd-Fe-B magnets and their corrosion products. The corrosion products were analyzed in the extracts of culture medium where the magnets had been immersed for 10 days at 37 ºC. Elements B, Co, Fe, La, Nd, Dy, Pr, Sm, Ho, Yb and Lu were found in the magnet and the analysis of extract indicated that Co, Fe and Nd are released from the magnet to the extract. Toxicity was also investigated in this extract using the neutral red uptake cytotoxicity assay.

KeyWordS: magnets, corrosion, Nd-Fe-B, cytotoxicity

I. INTRODUCTION

A special group of magnets composed mainly by Nd-Fe-B has been widely used in dental applications as retentive devices for overdentures, due to their strong force and compactness. Dental materials should present high corrosion resistance and be innocuous to human tissues, however, Nd-Fe-B magnets are highly susceptible to corrosion.

Kaszuwara and Leonowicz[1] performed long-term corrosion tests on Nd-Fe-B magnets and showed that the corrosion process proceeds faster for magnets containing higher concentrations of Nd and the resistance to atmospheric corrosion can be improved by adding Co, Al, Zr, C or Cu. Fernengel et al [2] also studied the effect of Co concentrations on the corrosion stability and they conclude that about 3.5 % Co addition are required for improved corrosion stability.

Therefore, there is a continuing concern improving the corrosion resistance of Nd-Fe-B magnets as well as on the study of the potentially harmful biological effects caused by toxic elements released from the magnet.

In this study, the elemental composition of a commercial sintered Nd-Fe-B magnet and of its corrosion products were evaluated. The toxicity of this sintered magnet was also investigated in the neutral red uptake cytotoxicity assay.

The method of inductively coupled plasma optical emission spectrometry (ICP-OES) was used to analyze B and instrumental neutron activation analysis was carried out for Co, Cr, Fe and lanthanide elements determinations.

II. EXPERIMENTAL

Sample of Nd-Fe-B magnet and its preparation for analysis. The magnet used in this study was a sintered commercial grade Nd-Fe-B supplied by CRUCIBLE Co., known as Crumax. Small chips of this magnet, with mass varying from 15 to 35 mg, were weighed in a clean polyethylene envelope for irradiation in the nuclear reactor.

INAA of the magnets. Samples and elemental standards were irradiated under thermal neutron flux of the IEA-R1 nuclear reactor for 30min and 8 h. After adequate decay times, the irradiated sample and standard were measured using a Model GX2020 hyperpure Ge detector coupled to Model 1510 Integrated Signal Processor and MCA System 100, both from Canberra (USA). Counting times of 200 s and of 5 to 8 hours were used, depending on the half lives or activities of the radioisotopes considered. The S100 software from Canberra was used to obtain gamma spectrum data that were processed using the VISPECT2 computer software[3]. The following radioisotopes were used: ^{60}Co, ^{52}Cr, ^{59}Fe, ^{140}La, ^{142}Pr, ^{147}Nd, ^{153}Sm, ^{165}Dy, ^{166}Ho, ^{169}Yb and ^{177}Lu. Concentrations of elements were calculated by comparative method.

ICP-OES of magnets. 1 mL of extract was taken and made to 10 mL with high purity water (18.3 MΩ cm^{-1}) Millipore German System. Nd, Fe and B were measured at 401.225 nm, 259.940 nm and 182.640 nm respectively, in an ICP-OES spectrometer (Spectroflame M120, Spectro, Germany). All reagents were analytical grade

Corrosion test. The surfaces of the magnet specimens were prepared by sequential grinding with silicon carbide

paper from grit #120 to #1000, and then they were immersed in a cell culture medium, minimum Eagle's medium (MEM), to evaluate their corrosion performance. The ratio surface area of magnet and volume of MEM was $1cm^2$ mL^{-1}. The extract is the final medium, after the time of immersion of the Nd-Fe-B. After a period of immersion of 48 hours and 10 days at 37 ^oC, the magnets were removed and the extracts were collected to study the toxicity and to analyze corrosion products.

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Cytotoxicity assay. The toxicity was investigated by a cytotoxicity test, carried out with the exposure of cell culture to the extract of the magnets. The cell line used was NCTC clone 929 from American Type Culture Collection (ATCC). The cytotoxic effect was evaluated using the neutral red uptake method, according to Ciapetti et al [4]. The negative control used was Ti, whose extract was processed in the same way of that used with the magnet, and the positive control consisted of phenol solution (0,02%), following the International Standard Organization (ISO) [5].

INAA of corrosion products. 500 µL of the extract solution were pipetted and dried in polyethylene capsules from Faculty of Biology, (Vie Universities, Amsterdam) for irradiation at the IEA-R nuclear reactor with elemental synthetic standards. These synthetic standards were prepared drying 50 µL of multielemental standard solutions in polyethylene capsules. Samples and standards were irradiated for 16 h with thermal neutron flux of 5 10^{12} cm^{-2} $s^{-1.}$ After adequate decay times, gamma ray measurements were carried out using hyperpure Ge detector. Blanks and culture media were also analyzed using the same experimental conditions adopted in the analysis of corrosion products extracts.

III. RESULTS AND DISCUSSION

The analysis of Nd-Fe-B showed that the elements B, Co, Fe, Nd, and Dy are present in this magnet at the levels of percentage and La, Pr, Sm, Ho, Yb and Lu at the µg g^{-1} levels (TABLE 1). Element Co is added to magnet for partial substitution of Fe by Co to improve the temperature coefficient of the remaining polarization. Nd-Fe-B magnet is also improved by a partial replacement of Nd by Dy which increases the anisotropy field strength and the intrinsic coercivity[2].

Analysis of extracts of Nd-Fe-B magnet showed corrosion of this material. TABLE 2 presents results of Co, Fe and Nd found in the extract of specimens submitted to the corrosion test.

As expected, results obtained in this work showed that sintered Nd-Fe-B magnet is corroded by the cell culture medium used.

In the cytotoxicity assay, after contact of NCTC L929 cell culture with serially dilution of 48 h and 10 days extract (100, 50, 25, 12.5 and 6.25%), the results showed a similar behavior to that of the negative control, that is, no cytotoxic effect. However, the cells in contact with the positive control showed a toxic effect, presenting a cytotoxicity index ($IC_{50\%}$) of approximately 12. The percentage of viability was calculated in relation to cell control (100%) and plotted in a graphic to obtain $IC_{50\%}$, as shown in Fig. 1.

TABLE 1. Elemental Composition of Sintered Nd-Fe-B Magnets Obtained by INAA and ICP-OES

Elements	Concentrations
B, %	0.7
Co, %	1.22 ± 0.04
Fe, %	57.35 ± 0.13
La, µg g^{-1}	41.3 ± 1.0
Pr, µg g^{-1}	878 ± 78
Nd, %	24.6 ± 0.2
Sm, µg g^{-1}	7.7 ± 1.1
Dy, %	2.126 ± 0.009
Ho, µg g^{-1}	5.94 ± 1.12
Yb, µg g^{-1}	1051 ± 32
Lu, µg g^{-1}	85.2 ± 2.1

TABLE 2. Elemental Concentrations in Culture Media After Corrosion Test of Nd-Fe-B and Results Obtained in Blank

Element	Blank of culture medium + capsule	Nd-Fe-B extract*
Cr, µg mL^{-1}	0.786 ± 0.007	0.601 ± 0.039
Co, µg mL^{-1}	0.0099 ± 0.0004	**2.92 ± 0.09**
Fe, µg mL^{-1}	Not detected	**85.3 ± 2.9**
Nd, µg mL^{-1}	Not detected	**25.9 ± 1.2**

*- Contribution from blank was discounted
The uncertainty of the result was evaluated using statistical counting errors of sample and standard.

IV. CONCLUSION

The Nd-Fe-B magnet presented no toxicity in the in vitro test of cytotoxicity. This type of magnet even though suffers corrosion in physiological medium can be used as biomaterials in an adequate capsule.

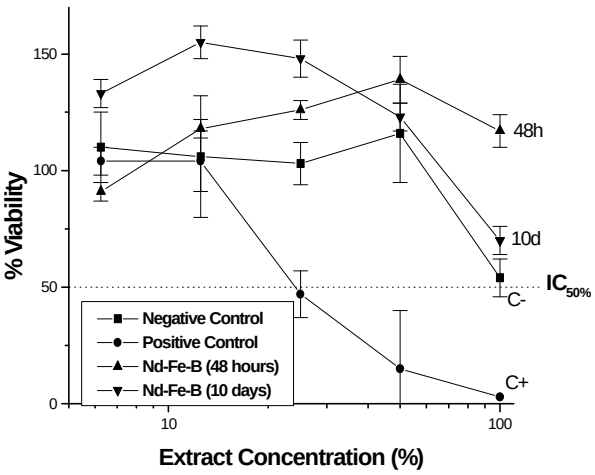

Figure 1. Cytotoxicity test. Viability Curves by Neutral Red Uptake Assay of Nd-Fe-B Magnet

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