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# Corrosion Performance and Cytotoxicity of Sintered Nd-Fe-B Magnets

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**Abstract:** In this investigation, the corrosion performance of a commercial sintered Nd-Fe-B magnet in a cell culture medium, and the cytotoxic response due to the magnet corrosion products leached into the cell culture medium, have been studied. Localized corrosion was observed and the corrosion products leached into cell culture medium were analyzed by ICP-OES. The magnet showed no toxicity in the neutral red uptake assay used in this investigation contradicting results from literature obtained by another method of cytotoxicity assay.

## Introduction

The use of magnets in dentistry has been reported since the 1950's [1]. Their main applications, in the past, were to improve retention and stability of dental prostheses. Magnets have been used for overdentures [2], dental implants [3] and maxillofacial prostheses [4]. However, the high cost of the magnetic materials and the large size required to produce adequate forces, have limited their widespread use [5]. Since the introduction of Rare Earth magnets, initially those based on samarium-cobalt, in the late 1960s [6], and then those based on neodymium-iron-boron (Nd-Fe-B), in the early 1980s [7], it has become possible to produce magnets with small dimensions enough to be used in dental applications. These magnets show great improvements in the maximum energy product comparatively to the old types, leading to a huge reduction in the size required to generate a particular magnetic flux [8]. Many studies on samarium-cobalt (Sm-Co) magnets as a dental material have been carried out since their development [9-10]. The maximum energy product of Sm-Co magnets is 33 MGOe whereas that of NdFeB magnets is in the range from 36 to 50 MGOe. These last magnets are increasingly replacing the Sm-Co types.

Nowadays, one of the widespread use of Nd-Fe-B magnets in dental applications is as retentive devices for overdentures, mainly due to their strong force and compactness. The dental materials should have high corrosion resistance and be innocuous to tissues. However, Nd-Fe-B magnets are highly susceptible to corrosion.

One of the main problems associated with the use of these magnets in clinical field is corrosion, and therefore, they are usually used encapsulated inside stainless steel or titanium cans. Another problem is the wear. Due to wear of the can or failure of the laser weld, saliva is able to leak into the can and cause corrosion of the magnet. This process can be stimulated by the presence of bacteria such as *Streptococcus sanguis* [5].

In this study, a commercial sintered Nd-Fe-B magnet was evaluated for cytotoxic response due to corrosion products.

## Material and Methods

The magnet used in this study was a sintered commercial grade Nd-Fe-B supplied by CRUCIBLE Co., known as Crumax. Its elemental composition was evaluated by the methods of instrumental neutron activation analysis (INAA) and the method of inductively coupled plasma optical emission spectrometry (ICP-OES). The results obtained, in percentages, were: B = 0.7; Co = 1.22; Fe = 58; Nd = 26.8.

The surfaces of the magnets 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 between the 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, the magnets surfaces were examined by scanning electron microscopy (SEM), the corroded area was analyzed by EDS and the respective extracts were collected to study their toxicity and to analyze corrosion products using ICP-OES method.

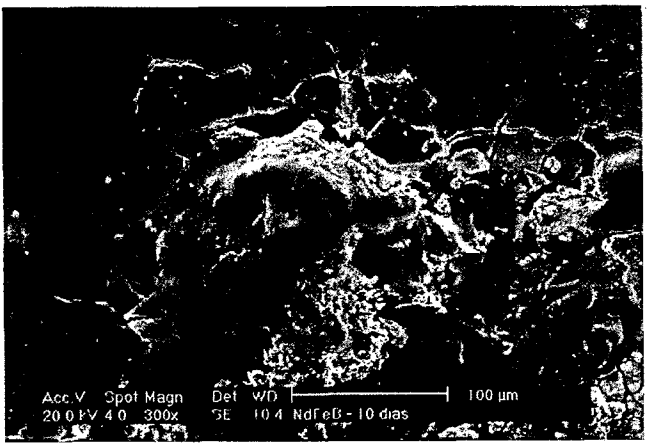
*NAA.* The experimental procedure adopted in the INAA of the magnet is described in a previous paper [11]. Small chips, mass varying from 15 to 35 mg, were weighted in a clean polyethylene envelope for irradiation with multielemental standard under thermal neutron flux of the IEA-R1m nuclear reactor, at IPEN. The induced gamma activities to the sample and standards were measured using a Ge detector coupled to a gamma ray spectrometer.

*ICP-OES.* 1 mL of extract was taken and diluted to 10 mL using high purity water (18.3 $M\Omega$ $cm^{-1}$) System, Germany). 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 the reagents were of analytical grade.

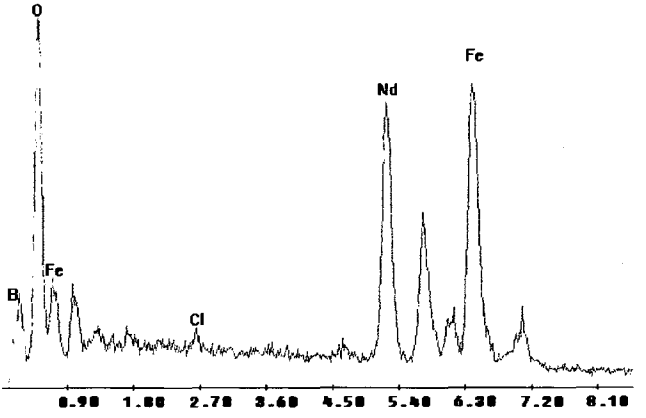
*Cytotoxicity assay:* The toxicity was investigated by a cytotoxicity test, carried out with the exposure of cell culture to the extract of the magnets. The cell line used was NCTC clone 929 from American Type Culture Collection (ATCC). The cytotoxic effect was evaluated using the neutral red uptake method, according to Ciapetti et al [12]. Ti was used as negative control and its extract was processed in the same way of that used for the magnet, and the positive control consisted of 0.02% phenol solution, according to the procedure of the International Standard Organization (ISO) [13].

## Results and Discussion

The magnets that remained immersed in the cell culture medium for 10 days, presented localized corrosion, such as pitting, and corrosion products with cracks, as Fig. 1 (a) shows. Localized corrosion to a lower extent was already found on the magnet after 24 and 48 hours of immersion in the culture medium, whose results are reported elsewhere [14]. EDS analysis of the corroded area, showed the presence of chlorine on it, Fig. 1 (b). The chloride ions in the cell culture medium might have led to corrosion initiation on active of the sintered magnet. Sintered magnets have pores in their microstructures and these are active areas more prone to corrosion. Besides they also have a complex microstructure made of a mixture of phases (Nd–rich phase and the magnetic ($\Phi$) phase) of very distinct electrochemical behavior. The combination of porosity/complex microstructure and chloride ions are most likely the cause of the localized corrosion, as pitting, observed in the Nd-Fe-B magnet tested.




(a)




(b) λ, Energy

Fig. 1. (a) Localized corrosion on Nd-Fe-B magnet after 10 days of immersion in MEM . (b) EDS spectrum on the corroded area.

The results obtained in the analyses of corrosion products, presented in Table 1, show that Nd-Fe-B magnets are corroded when exposed to the culture medium, as expected, and this corrosion process continued along the time.

Table 1. Elemental concentrations obtained in the analyses of extracts with corrosion products.

| Element | Method: ICP-OES | |
|---|---|---|
| | 48 h immersion | 10 d immersion |
| B, µg $mL^{-1}$ | 1.544 | 3.74 |
| Fe, µg $mL^{-1}$ | 1.39 | 37.48 |
| Nd, µg $mL^{-1}$ | 0.21 | 1.52 |

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 these data were plotted in a graphic to obtain $IC_{50\%}$, as shown in Fig. 2.

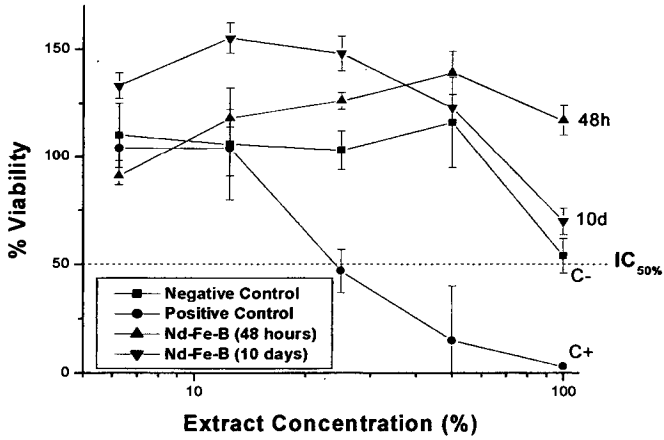



Fig. 2: Cytotoxicity test. Viability curves by neutral red uptake assay of Nd-Fe-B magnet.

Results obtained using *in vitro* cytotoxicity test with modified agar overlay technique, presented by Donohue et al [15], revelead that Nd-Fe-B magnets present toxic effect. This technique uses indirect contact of the material with cell culture. The neutral red uptake assay also uses the indirect contact technique, but with the exposure of extract of the material to cell culture. It is likely that the toxic elements leached from the magnets in the overlay technique cross the agar layer and damage the cells, during 24 h of contact. When the toxic elements are leached into the cell culture medium during 48 h and 10 days, they can react with some anions of the medium like phosphate and they form precipitates, that does not cause cytotoxic effect in the neutral red uptake assay Therefore this subject deserves further investigation and the study is continuing in order to clarify these contradictory results.

## Conclusions

In this investigation Nd-Fe-B sintered magnets (Crumax) showed localized corrosion after their immersion in a cell culture medium (MEM). The corrosion products presented no cytotoxicity in the neutral red uptake assay, contradicting literature results obtained by other method of cytotoxicity assay. Further investigation has to be done to use safely these kinds of magnets as implant.

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**References**

[1] H. Freedman, J.Am. Dent. Assoc. 47 (1953), p. 288.
[2] B.K. Moghadam and F.R. Scandrett, J.Prosthet.Dent. 41 (1979), p. 26.
[3] S.J. Behrman, J. Am. Dent. Associ. 68 (1964), p. 206.
[4] N. Javid, J. Prosthet. Dent. 25 (1971), p. 334.
[5] M. Wilson, H. Kpendema, J.H. Noar, N. Hunt and N.J. Mordan, Biomaterials 16 (1999), p. 721.
[6] J. Becker, Scientific American 223 (1970), p. 92.
[7] M. Sagawa, S. Fugimura, N. Togawa, H. Yamamoto and Y. Matsuura, J. Appl. Phys. 55(6) (1984), p. 2088.
[8] I.R. Harris, Mater Sci. Technol. 6 (1990), p.962.
[9] H. Sasaki, Y. Kinouchi, Y. Yoshida, H. Tsutsui and T. Ushita, Proceedings of the Fourth International Workshop on Rare Earth-Cobalt Permanent Magnets and Their Applications. Tokyo, 1979, Society of Nontraditional Technology, p. 83.
[10] H. Tsutsui, Y. Kinouchi, H. Sasaki, M. Shiota and T. Ushita, J. Dent. Res. 58 (6) (1979), p. 1597.
[11] M. Saiki, S.O. Rogero, I. Costa, O.V. Correa, O.Z. Higa, J. Radioanal. Nucl. Chem. 248 (1) (2001), p. 133.
[12] G. Ciapetti, D. Granchi, E. Verri, L. Savarino, D. Cavedagna, A. Pizzoferrato, Biomaterials 17 (1996),p.1259.
[13] ISO document 10993-5, 1992 Biological evaluation of medical devices, Part 5, Tests for cytotoxicity: in vitro methods.
[14] S.O. Rogero, M.C.L. Oliveira, M. Saiki and I. Costa, SEM of corroded NdFeB magnets in a culture medium, submitted to Acta Microscopica.
[15] V.E. Donohue, F. MacDonald, R. Evans, J.Appl.Biomat. 6(1) (1995), p. 69.