Studies About Degradation of Zirconia in Artificial Saliva

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To study zirconia degradation, we used samples with 16.5 mm diameter and 3 mm thick, milled from IPS e.max Zir CAD disks and subjected to sintering. Samples were immersed in Fusayama Meyer artificial saliva, removed after one day, 3 days, 7 days, 14 days and 21 days and examined from the point of view of surface morphology and elemental composition with an electronic scanning microscope. By SEM examining of samples prior to immersion in artificial saliva we found that the material contained strictly the chemical elements specified by the manufacturer. SEM examination and mass evaluations performed after immersion in artificial saliva demonstrate that the material did not suffer degradation processes, the minimum differences in the measurements being within the analytical balance weighing range.

Keywords: zirconia, artificial saliva, degradation

An in vitro study [4] which investigating the effect of aging on zirconia used in oral rehabilitation has found that, although the aging process reduces the mechanical characteristics of zirconia, the decrease occurs within clinically acceptable values.

To assess the degree of degradation of ceramics in different solutions, it is not possible to use electrochemical techniques, it is preferable to immerse the samples under similar conditions to the environment in which the materials are used.

Experimental part

To study zirconia degradation, we used samples with 16.5 mm diameter and 3 mm thick, milled from IPS e.max Zir CAD disks and subjected to sintering.

Samples were immersed in Fusayama Meyer artificial saliva (0.4 gl⁻¹NaCl, 0.9 gl⁻¹KCl, 1 gl⁻¹uree, 0.69 gl⁻¹NaH₂PO₄, 0.795 gl⁻¹CaCl₂⋅2H₂O) at pH 5.2 maintained at 37 ± 0.1 °C by a Memmert IF55 incubator.

Zirconia samples were examined before immersion in artificial saliva from the point of view of surface morphology and elemental composition with an electronic scanning microscope (SEM) equipped with an energy dispersion spectrometer - EDS (Phenom ProX model, manufacturer from PhenomWorld, Netherlands).

Before the immersion tests were performed, the samples were sonicated for 20 min in acetone to be degreased and cleaned with impurities and then rinsed with ultrapure water (ASTM I type).

We used 5 discs to track their mass in pre-established time ranges: one day, 3 days, 7 days, 14 days and 21 days. For this purpose we used a Kern ALT 100-5AM balance with an accuracy of 0.01 mg.

Samples were removed from artificial saliva at predetermined ranges, cleaned with deionized water, dried for one hour in a Memmert UF55 oven, and then stored 72

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hours in the desiccator before being weighed. Their mass measurements after the immersion tests were repeated 5 times for the best accuracy of the results.

Results and discussions

The elemental composition obtained by analyzing one of the zirconia disks is shown in table 1, and figure 2 illustrates the EDS spectrum corresponding to the compositional analysis.

Images of zirconia surface morphology at different magnification powers can be found in figure 3.

Table 1

| No. | Element | Concentration |
|-----|---------|---------------|
| 1   | O       | 59.59 %       |
| 2   | Zr      | 35.08 %       |
| 3   | Y       | 3.45 %        |
| 4   | Hf      | 1.36 %        |
| 5   | Al      | 0.52 %        |

From the SEM images it is observed that after sintering, the sample surface morphology is composed of zirconia particles, with small defects of compactness. The particle size of ZrO₂ is between 1 ÷ 3 µm.

The results on the mass evolution of the samples tested for degradation by immersion in artificial saliva in all predefined time intervals are centralized in table 2. We find the presence of very small differences, ranging from 0.01 to 0.02 mg, which are in the analytical balance error range. Thus, it can be concluded that zirconia in sintered form does not undergo degradation processes in artificial saliva even after 21 days of immersion.

Following the immersion tests, the samples were investigated using the Phenom ProX electronic scanning microscope for a surface analysis to identify possible changes to the original morphology. The SEM images of the samples after the immersion test are shown in figures 4-8.

Among the materials widely used in general medicine and dentistry, zirconia combines high resistance, remarkable resistance to crack propagation, low thermal conductivity, high ionic conductivity, biocompatibility and chemical inertia [3, 5-8]. The transformation of the tetragonal phase into the monoclinic in the zirconia [9] results in increased resistance to degradation and increase of fracture resistance [6] of the material. This transformation mechanism makes zirconium the strongest and most fracture resistant material of all ceramic biostructures [10-12].

Over the last thirty years, zirconia has been used in restorative dentistry for the manufacture of dental implants and abutments [13]. Over the past twenty years, zirconia has been used as a substrate for making crowns and dental bridges in the lateral area [5, 6, 10, 14]. Recently, colored zirconia and improved translucency have been developed to have a close look at human dentition [15]. This new material has a bending strength of 900-1400 MPa and a breaking strength of up to 6 MPa m1/2 [16]. Such advantages have led to an exponential increase in the use of zirconia for monolithic crowns and dental bridges in the posterior region [17] of the dental arches.

Choosing an ideal aesthetic material for dental restorations is a major desideratum in dentistry and necessity for improvement of the materials has led to a significant change in available materials and usage techniques [18].

In the case of use of zirconia for making medical and dental devices, the material must be treated on the surface to obtain not only mechanical functions such as wear...
resistance but also biomedical capabilities such as cell adhesion and bacterial decolonization [15].

Conclusions

By SEM examining of samples prior to immersion in artificial saliva we found that the material contained strictly the chemical elements specified by the manufacturer. SEM examination and mass evaluations performed after immersion in artificial saliva demonstrate that the material did not suffer degradation processes, the minimum differences in the measurements being within the analytical balance weighing range.

References

1. Osman RB, Swain MV. A Critical Review of Dental Implant Materials with an Emphasis on Titanium versus Zirconia. Materials 2015, 8, 932-958; doi:10.3390/ma8030932.
2. Lawson S. Environmental degradation of zirconia ceramics. J. Eur. Ceram. Soc. 1995, 15, 485–502.
3. Chevalier J. What future for zirconia as a biomaterial? Biomaterials 2006, 27, 535–543.
4. Att W, Grigoriadou M, Strub JR. ZrO2 three-unit fixed partial dentures: Comparison of failure load before and after exposure to a mastication simulator. J. Oral Rehabil. 2007, 34, 282-290.
5. Miyazaki T, Nakamura T, Matsushima H, Ban S, Kobayashi T. Current status of zirconia restoration. J Prosthodont Res 2013;57:236-61.
6. Denry I, Kelly J. R. State of the art of zirconia for dental applications, Dent. Mater., Vol. 24, No. 3, p. 299 - 307, ISSN 0109-564, 2008.
7. Abduo J, Lyons K, Swain M. Fit of zirconia fixed partial denture: a systematic review. J Rehabil 2010;37:866-876.
8. Yin L, Song XF, Song YL, Huang T, Li J. An overview of in vitro abrasive finishing and CAD/CAM of bioceramics in restorative dentistry. Int J Mach Tool Manu 2006;46:1013-1026.
9. Garvie RC, Nicholson PS. Phase analysis in zirconia systems. J Am Ceram Soc 1972;55:303-305.
10. Denry I, Kelly J. R. Emerging ceramic-based materials for dentistry. J Dent Res 2014;93:1235-42.
11. Chevalier J, Gremillard L, Virkar AV, Clarke DR. The tetragonal-monoclinic transformation in zirconia: lessons learnt and future trends. J Am Ceram Soc 2009;92:1901-1920.
12. Evans AG, Cannon RM. Toughening of brittle solids by martensitic transformations. Acta Met 1980;34:761-800.
13. Manicone PF, Iommetti PR, Raffaelli L. An overview of zirconia ceramics: basic properties and clinicla applications. J Dent 2007;35:819-826.
14. Ban S. Reliability and properties of core materials for all-ceramic dental restorations. Jpn Dent Sci Rev 2008;44:3-21.
15. Yin L, Nakanimshib Y, Aologna AR, Song XF, Abduo J, Zhang Y. A review of engineered zirconia surfaces in biomedical applications, Available online at www.sciencedirect.com, doi: 10.1016/j.procir.2017.04.057, Procedia CIRP 65 (2017) 284 - 290.
16. Zhang Y. Making yttria-stabilized tetragonal zirconia translucent. Dent Mater 2013; 29: 1201-1208.
17. Zhang Y, Lee JJW, Srikanth R, Law BR. Edge chipping and resistance of monolithic ceramics. Dent Mater 2013; 29: 1201-1208.
18. Earar, K., Grigoriou, R., Scutariu, M.M., Vasile, E., Antoniac, A., Dragomir, L., Gradinaru, S. Effect of the Sandblasting Process on the Surface Properties of Dental Zirconia, Rev Chim. (Bucharest), 68, no. 7, 2017, p. 1560-1564.

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