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Microstructural characterization and comparative evaluation of physical, mechanical and biological properties of three ceramics for metal–ceramic restorations

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ABSTRACT

Objectives. A wide variety of dental ceramics compositions have been introduced in dental clinical practice in order to combine desired aesthetics with superior mechanical performance. The aim of the present study was to investigate the microstructural changes in three dental ceramics after their sintering according to manufacturers' instructions and to comparatively evaluate some of their physical, mechanical and biological properties.

Methods. The analysis of the phases present in each material before and after sintering was performed with scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction analysis (XRD). The thermal properties of ceramic specimens were evaluated with differential thermal and thermogravimetric analysis (TG-DTA). The mechanical properties evaluated were fracture toughness, Young's modulus and microhardness with the Vickers indentation method. MTT assay was used for cell proliferation assessment. One-way analysis of variance (ANOVA) with Bonferroni multiple comparisons tests was used to determine statistically significant differences (significance level of $p < 0.05$).

Results. Results showed a remarkable variation among the three ceramic compositions of leucite content in the starting unheated ceramic powders ranging between 14 and 32 wt.% and in the respective sintered powders ranging between 15 and 41 wt.% The low fusing glass–ceramic and the high fusing leucite-based ceramic presented significantly higher fracture toughness ($p < 0.001$) and microhardness and lower modulus of elasticity ($p < 0.05$) compared to the low fusing feldspathic ceramic. The three ceramics were almost equivalent concerning their in vitro biological behavior.

Significance. Variations in crystal structure, distribution and composition are related to differences concerning mechanical properties of dental ceramics.

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1. Introduction

Dental ceramics at various compositions for metal–ceramic restorations were introduced in 1962 [1]. Since then, most of these ceramics are composed of leucite crystals dispersed in a glassy matrix. Leucite has a high coefficient of thermal expansion (CTE) and raises the overall thermal expansion of the bulk porcelain leading to thermal compatibility with metal frameworks [2,3]. The amount of leucite and the amount and composition of glass determine decisively the CTE of the final product.

Leucite structure relies on a framework of SiO_4 tetrahedra that form rings [4]. At room temperature leucite crystals are of tetragonal symmetry. When heated the SiO_4 structure expands slowly by untilting and untwisting the tetragonal rings until the symmetry changes to cubic at 625 °C [5,6]. The phase transformation of leucite from cubic to tetragonal when cooled, in combination with the greater contraction of the leucite crystals compared to the glassy matrix due to their large thermal contraction mismatch, cause tangential compressive stresses around leucite crystals [5]. These stresses may act either as crack deflectors or crack initiators influencing the mechanical performance of the ceramic [7,8].

The leucite content in dental ceramics is critical also due to its contribution to the flexural strength of feldspathic porcelains. It has been reported that an increase in leucite content from 10 to 30% increased the flexural strength from 34.1 to 64.8 MPa [9] while studies on fracture toughness (K_{IC}) showed a direct relation of the leucite content to K_{IC} [10,11]. Moreover, Cesar et al. [3] proved evidence that ceramic compositions with higher leucite content presented higher incidence of crack deflection. Commercial leucite dental ceramic compositions contain 17–45% of tetragonal leucite [12,13], although leucite volume fraction may be altered due to repeated firings or different cooling rates [3,13].

Glass–ceramics that can be sintered to conventional or fine metal frameworks have been introduced in clinical practice. They are polycrystalline solids prepared by the controlled crystallization of glasses [14] and have been reported to present improved optical, physicochemical or mechanical properties [15,16]. Glass–ceramics are made by forming special base glasses, mostly by melting, and then using controlled heat treatment (ceraming) to nucleate and precipitate crystals in the glassy matrix [17]. The number of crystals, their growth rate and thus their size are regulated by the time and temperature of the ceraming process. The chemical composition and microstructure of the glass–ceramic determine its properties and main applications, while to ensure high mechanical performance it is important that the crystals are numerous and uniformly distributed throughout the glassy phase [17]. Recently combined leucite-fluorapatite or fluorapatite glass–ceramics have been introduced in dental market, as fluorapatite crystals present increased chemical durability compared to that of natural teeth (hydroxyapatite), and closely resemble the crystals in natural teeth optimizing the optical properties of the material [15].

A major recent change in dental ceramic formulations for metal–ceramic restorations comprise the low fusing ceramics with about 200 °C lower fusion temperature compared to conventional dental ceramics. The reduction of fusion temperatures initially occurred in order to eliminate the problems of bonding between titanium and its alloys and dental ceramics. The exposure of the Ti alloy to temperatures that exceed 800 °C leads to the absorption of oxygen and nitrogen, providing the formation of a thick superficial layer of Ti oxide that may attain a thickness up to 1 mm and harms the bonding of ceramic to substrate [18,19]. In compliance with these criteria, additional clinical interest for these low fusing compositions arises from their significant advantages, as the reduction of the fusion temperature allows for increased opalescence, highly polished surface, and considerably less potential for abrading any materials against which they occlude [20].

However, there is limited literature concerning the physicochemical properties of the wide range of dental ceramics used in clinical practice. Microstructural analysis is important because it provides an association among the composition, physical properties, and structural characteristics of the materials [21]. Even fewer studies [22–24] focus on the biocompatibility or cytotoxicity of these commercially available dental ceramics although they perform in direct contact with surrounding periodontal tissues. The biocompatibility of dental ceramics has been based on studies of traditional feldspathic porcelains, although many new materials are very different in composition [25,26].

The aim of this study was to identify the crystal phases of three representative leucite-based dental ceramics used in fixed prosthetic metal–ceramic restorations and to comparatively evaluate some of their physical, mechanical and biological properties. The ceramics were selected due to their different fusion temperatures and compositions. Furthermore, aspects of structure/property relationships will be investigated and discussed.

2. Materials and methods

The experimental procedures included the fabrication of ceramic specimens, the evaluation of their thermal properties, the identification and qualitative determination of their crystal structure, the evaluation of their mechanical properties and finally the evaluation of their biocompatibility. In details:

2.1. *Fabrication of ceramic specimens*

Ceramic disks of three commercial dental ceramics used in metal ceramic restorations (Table 1) were fabricated according to a procedure described elsewhere [27]. In brief, ceramic powder was mixed with modelling liquid using the same liquid to powder ratio (0.335) for the different ceramics, transferred into polysiloxane (Optosil®-Xantopren®, Heraeus Kulzer GmbH & Co. KG, Germany) moulds and condensed using a vibrator, to accomplish water removal. The disks were removed by gentle hand pressure and sintered (Programat P95), according to manufacturers' instructions (Table 1).