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Research Paper

Characterization of the bioactive and mechanical behavior of dental ceramic/sol–gel derived bioactive glass mixtures

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ABSTRACT

Dental ceramics can be modified by bioactive glasses in order to develop apatite layer on their surface. One of the benefits of such modification is to prolong the lifetime of the fixed dental prosthesis by preventing the formation of secondary caries. Dental ceramic/sol–gel derived bioactive glass mixture is one of the options for this modification. In the current study, mixtures of dental ceramic/bioactive glass with different compositions were successfully produced. To evaluate their bioactive behavior, prepared samples were immersed in a simulated body fluid at various time intervals. The prepared and soaked specimens were characterized using Fourier transform infrared spectroscopy, X-ray diffractometry and scanning electron microscopy. Since bioactive glasses have deleterious effects on the mechanical properties of dental ceramics, 3-point bending tests were used to evaluate the flexural strength, flexural strain, tangent modulus of elasticity and Weibull modulus of the specimens in order to find the optimal relationship between mechanical and bioactive properties.

1. Introduction

Ceramics are the most acceptable tooth colored restorative materials providing the best esthetics, in spite of being brittle and weak in tension (Sakaguchi and Powers, 2012). As ideal materials to be used in the harsh oral cavity for long periods of time, dental ceramics (DC) need to be compatible with tooth structure in terms of having high fracture strength and wear resistance (Höland et al., 2009). An application of these materials is in fixed prosthetic restorations. However, existence of a marginal gap between the tooth and restoration, being exposed to oral bacteria, results in pulp irritation or necrosis, secondary caries and cement dissolution, all being the common reasons of fixed prosthetic restoration failure (Wickens, 1999). If these ceramics were modified in a way that they could stimulate bioactive behavior around the

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restoration margins, they could develop periodontal tissue attachment and create complete sealing of the marginal gap. Sealing the gap may prevent the failure of fixed ceramic restorations by eliminating secondary caries, micropenetration of the oral bacteria and their adhesion on the marginal area (Craig and LeGeros, 1999). As cementum consists of biological hydroxyapatite (HA), the bioactive behavior can be stimulated by formation of apatite on the dental ceramic surface, providing the biological surface required for attachment of the cells (Kokoti et al., 2001).

The most suitable candidate for achieving bioactive behavior is bioactive glass since the ions released can induce osteogenic differentiation even without contact to bioactive glass (Ojansivu et al., 2015). Furthermore, bioactive glasses are capable of increasing the pH of the interfacial solution through the dissolution of ions resulting in a change in ion concentration. This in turn affects the viability of a several bacterial species (Vallittu et al., 2015).

Various studies have been conducted in order to develop apatite on the dental ceramic surfaces through modification with bioactive glasses (BG) (Abbasi et al., 2015). In 2003, a dental ceramic was coated by bioactive glass, and the growth of a well-attached apatite layer on the surface was observed after immersion in simulated body fluid (Papadopoulou et al., 2003). Moreover, it was reported that the attachment and proliferation of human periodontal ligament cells can be supported by dental ceramic/bioactive glass mixtures (Kontonasaki et al., 2003). Since 2010, many researchers have anticipated to produce sol–gel derived dental ceramic/bioactive glass mixtures (Chatzistavrou et al., 2010; Goudouri et al., 2011a, 2011b; Manda et al., 2012; Chatzistavrou et al., 2012; Goudouri et al., 2014). Sol–gel method provides a better control of composition, microstructure and properties due to high homogeneity, compared to melt-derived ceramics (Chatzistavrou et al., 2010). Therefore, they utilized sol–gel method to produce bioactive glass, and during the process, they added different amounts of dental ceramics (only used for metal-ceramic restorations) to create DC/BG mixtures (Goudouri et al., 2011a, 2011b; Manda et al., 2012; Chatzistavrou et al., 2012; Goudouri et al., 2014).

Therefore, the aim of this study was to prepare a sol–gel derived mixture of a commercial dental ceramic (IPS e.max used for all-ceramic restorations) and a bioactive glass (58S) in order to provide apatite formation ability for the dental ceramic without compromising its mechanical properties. In this way, the chemical composition, textural properties and bioactivity, as well as mechanical properties of the mixture such as flexural strength, flexural strain, tangent modulus of elasticity and Weibull modulus, have been investigated.

2. Materials and methods

Sol-gel-derived bioactive glass (58S) was produced as described in the literature (Zhong and Greenspan, 2000). In details, 1.6 ml nitric acid (Merck, Darmstadt, Germany) was gradually added into 9.5 ml DI water and then 13.2 ml tetraethoxysilane (TEOS, $Si(OC_2H_5)_4$, Acros Organics, New Jersey, USA) was poured into the mixture. After 30 min of mixing, 0.7 ml triethylphosphate (TEP, $OP(OC_2H_5)$, Fluka Chemika, Switzerland) was added to the mixture. After 20 min of mixing, 5.8 g calcium nitrate tetrahydrate (CN, $Ca(NO_3)_2 \cdot 4H_2O$, Merck, Darmstadt, Germany) was added to the mixture. The mixing process continued for 1 h to complete the dissolution of calcium nitrate and continue the hydrolysis reaction. Various amounts of dental ceramic powder, used for all-ceramic restorations (IPS e.max-dentin, Ivoclar, Schaan, Liechtenstein) [70, 60, 50 and 40 wt%], were added to 58S in solution stage, before the aging and drying processes. The mixtures were stirred continuously until gelatation. The drying process was conducted in an oven at 180 °C for 6 h followed by the stabilization cycle in a furnace for 18 h at 700 °C. The products were pulverized in a ceramic mortar and sieved to powders (particle size <40 μm).

All prepared powder mixtures as well as pure 58S and pure dental ceramic powders were examined by Fourier Transform Infrared spectroscopy (FTIR; Spectrum RXI, Perkin-Elmer, Massachusetts, USA) and X-ray Diffractometry (XRD; D8 Advance, Bruker, Massachusetts, USA). FTIR analysis performed in transmittance mode in mid-infrared range of 400–4000 cm^{-1}. The samples were also analyzed by XRD with CuKα radiation of 40 kV and 40 mA. Data were obtained at the scan speed of 0.01° 2θ $s^{-1}$and the step size of 0.05° 2θ in the 2θ range of 10–70°.

The apatite-forming ability of the powders was tested through immersion in simulated body fluid (SBF) (Kokubo and Takadama, 2006) with the constant powder to solution ratio of 50 mg/75 ml at ~37 °C. Immersion in SBF was carried out for 6 h, 1, 3, 6 and 9 days. Every three days, the SBF solution was replaced to avoid the large reduction of cation concentration in the solution. After removal of the powders from SBF, they were rinsed with ethanol and distilled water, dried and stored in airtight containers.

The reacted powders were examined by FTIR and XRD, with previously described parameters, to analyze their bioactive behavior through the assessment of the possible formation of an apatite layer on the powder surface. Scanning electron microscopy (SEM; S360, Leica Cambridge Ltd., Cambridge, UK) was also used for the microstructural characterization of the samples after they were coated with gold in a sputter coater (SC7640, Polaron, East Sussex, UK).

According to ISO 6872 (2008), the rectangular specimens were fabricated from all powder samples. The sieved powders were mixed with distilled water to obtain slurries which were transferred in a mold and condensed using a vibrator to remove water. Then, they were removed from the mold by gentle hand pressure and heat treated according to the manufacturer's instructions in a furnace appropriate for dental materials (Programat® P700). Using optical microscopy, ten flawless specimens of each composition were selected. The samples were loaded at a crosshead speed of 0.5 mm/min using a universal testing machine (Zwick/Roll Z020; Zwick GmbH & Co, Germany). For 3-point bending test, the maximum load at specimen failure was recorded and the flexural strength was calculated using Eq. (1).

$$\sigma = \frac{3Pl}{2wb^2}, \quad (1)$$

where P is the ultimate load at fracture, l is the distance of the supports, w is the width of the specimen and b is the