Research Article

Dental Ceramics/Bioactive Glass Composites: Characterization and Mechanical Properties Investigation

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Abstract Apart from a good bioactive behavior, composite materials for dental applications should attain high mechanical properties. Thus, the aim of this work was the fabrication and characterization of novel sol-gel derived dental ceramics/bioactive glass composites and the investigation of their mechanical properties such as flexural strength and Weibull modulus. Fourier Transform Infrared Spectroscopy (FTIR) and Surface Scanning X-ray Diffractometry (XRD) revealed the crystallization of leucite. Flexural strength values were in the range of 5–50 MPa and it was followed a linear relationship between the amount of dental porcelain in the composites and the strength of the materials. In conclusion, dental ceramics/bioactive glass composites exhibit mechanical integrity and can be potentially used in restorative dentistry.

Keywords sol-gel; 58S bioglass; dental ceramic; flexural strength; Weibull modulus; FTIR, XRD

1 Introduction

Bioactive dental ceramics could enhance periodontal tissue regeneration around the margins of fixed prosthetic restorations, eliminating the marginal gap between tooth and fixed prosthesis which is responsible for cement dissolution, secondary carries and eventually the failure of the restoration. The fabrication of dental ceramics/bioactive glass composites (DC/BG) able to elicit bioactive behavior has been previously reported [2,3]. Furthermore, Chatzistavrou et al. [1], reported the fabrication of two novel sol-gel derived materials for dental applications; a glass-ceramic and a bioactive composite material (glass-ceramic 30 wt%bioactive glass 58S 70 wt%) with characteristics similar to that of a commercial dental ceramic, which both exhibited good control of composition, microstructure and properties, equivalent in vitro biological behavior and presented increased rate of cell proliferation after the 3rd day of cultivation period. However, apart from a good bioactive behavior, composite materials for dental applications should attain high mechanical properties. Flexural forces are the result of forces generated in clinical situations and the dental materials need to withstand repeated flexing, bending, and twisting. A high flexural strength is desired once these materials are under the action of chewing stress that might induce permanent deformation [9]. The minimum flexural strength of dental ceramics intended to be used as aesthetic ceramics for coverage of a metal or a ceramic substructure is 50 MPa [5]. Ceramic strength data are generally not normally distributed around the mean, but often skewed in the high strength portion. In contrast to the mean value, the Weibull modulus compensates for this lower range of values whose asymmetry is typical for ceramic materials [9, 8]. Hence, it is clear that Weibull analysis of strength data approaches better the fracture potential of these ceramic materials. A large value of Weibull modulus ensures fewer fatal flaws, a smaller error in strength estimation, and greater clinical reliability [8]. Thus, the aim of this work was the fabrication and characterization of novel sol-gel derived dental ceramics/bioactive glass composites and the investigation of their mechanical properties such as flexural strength and Weibull modulus.

2 Materials and methods

Dental ceramics/bioactive glass composites were prepared by the sol-gel method [10], while during the gelation an appropriate quantity (50–70% wt ratio) of a dental ceramic were added in the mixture. The dental ceramic used was a leucite based porcelain (IPS InLine®, Ivoclar, Schaan, Liechtenstein). The resulting mixture was sieved to a powder of $< 40\,\mu$m and was mixed with distilled water. The resultant slurry was transferred in polyethylene molds in order to fabricate rectangular specimens according to
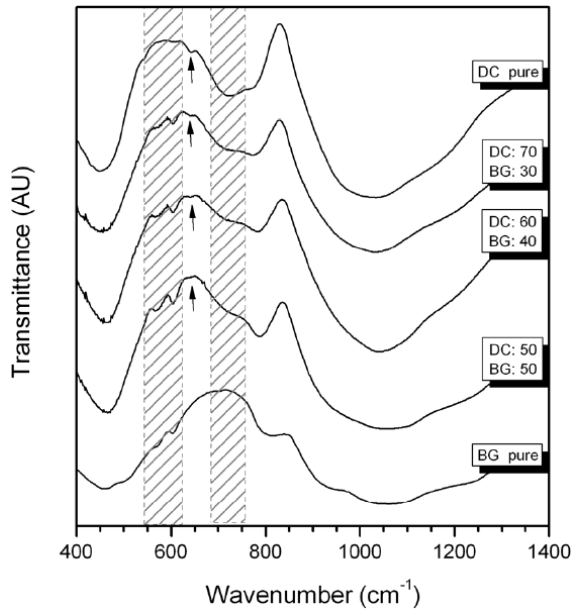
Figure 1: FTIR spectra of all composite materials, pure bioactive glass and pure dental ceramic.

ISO 6872 [5], which were heat treated at 930 °C according to manufacturer's instructions [6]. The specimens were observed with an optical microscope and those with flaws were excluded. Ten flawless specimens of each category were immediately loaded at a universal testing machine for the 3-point bending test (Instron 3344) with a cross-head speed of 0.5 mm/min until break. Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffractometry (XRD) were used to characterize all specimens. The FTIR transmittance spectra by the KBr pellet technique were obtained using a Perkin-Elmer Spectrometer Spectrum 1000 in MIR region with a resolution $4\,cm^{-1}$.

The XRD measurements were carried out by using a Philips (PW1710) diffractometer with Ni-filtered CuKα radiation. The counting statistics of the XRD study were: step size 0.05° 2θ, start angle 5°, end angle: 75° and scan speed: 0.01° 2θ/sec.

The load at yield is the sample material's flexural strength that is calculated by the following formula: $\sigma = 3Pl/wb^2$, 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 thickness of the specimen. "One-way ANOVA and Bonferrroni's multiple comparison tests were used to determine whether differences in group means were statistically significant at $p < 0.05$. The Weibull modulus and characteristic strength were estimated from flexure strength data by rank order statistics. The strengths from thirty specimens in ascending order were ranked and each specimen was assigned with a probability of failure based on its ranking, described by the following formula:
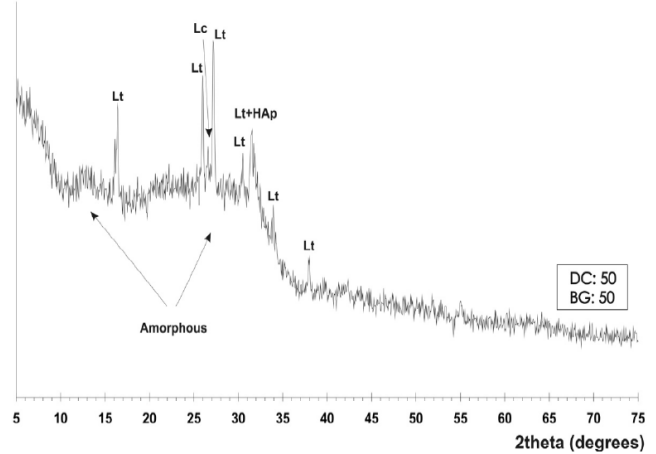

Figure 2: XRD pattern of 50:50 DC/BG composite.

Pf $= (i - 0.5)/N$, where i is the $1, 2, 3, 4, \ldots, i$th; N is the number of specimens in batch. The variables Pf and σ were transformed to $\ln \ln[(1/1 - Pf)]$ and $\ln \sigma$, respectively and a plot was constructed with $\ln \ln[(1/1 - Pf)]$ on the ordinate and a corresponding $\ln \sigma$ on the abscissa where the slope of the curve is equal to Weibul modulus (m)" [5].

3 Results and discussion

The FTIR spectra (Figure 1) of all heated specimens prove the coexistence of the two constituents in the mixture by revealing the characteristic bands of both bioactive glass and dental ceramic.

In details, the broad peak at $1100\,cm^{-1}$ and the peak at $796\,cm^{-1}$ assigned to the asymmetric and symmetric Si-O-Si stretching vibration modes, respectively, and the peak at $450\,cm^{-1}$ that is attributed to the Si-O bending mode [3] are present in all spectra and attributed to the Si-Al network. Additionally, the presence of a weak double peak at 568 and $604\,cm^{-1}$ in all spectra, in which BG participates, is attributed to the bending of P-O mode revealing the crystallization of a Ca-P phase [2]. This finding is further confirmed by the small quantity of hydroxyapatite (HAp) that is detected in XRD patterns (Figure 2). Generally, SiO_2-CaO-P_2O_5 sol gel systems are prone to form various Ca-P [7] or Ca-Si phases [3]. The presence of the dental ceramic's grains during the formation of the DC/BG composites probably provides sites of heterogeneous nucleation facilitating the crystallization of the detected apatite phase. Finally, the peak at 714 and the weak peak at $642\,cm^{-1}$ are assigned to leucite ($KAlSi_2O_6$, Lt), which is a dominant phase in dental porcelains [3,6].

The intensity of the peak at $714\,cm^{-1}$ is linearly related to the amount of the dental ceramic in the composite. XRD patterns of all samples reveal the crystallization of leucite and a characteristic pattern is presented in Figure 2.
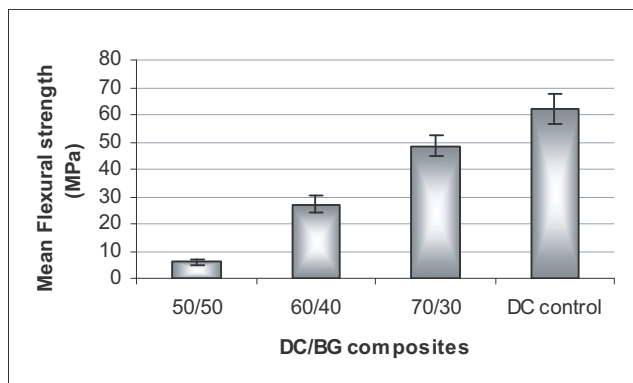
Figure 3: Mean values of flexural strength of control DC and DC/BG composite specimens.

Figure 4: Weibull's modulus composite specimens.

Mean values of flexural strength for the composite materials and the pure dental ceramic are presented in Figure 3. Control DC specimens showed the highest mean value (62.05 MPa), while an apparent and almost linear increase of the composites flexural strength was recorded as the dental ceramic percentage was increased.

The highest strength values among the composite materials were found for the 70/30 DC/BG composite (48.5 MPa) followed by 60/40 DC/BG composite (27.0 MPa) and 50/50 DC/BG composite (6.0 MPa). Bonferroni's multiple comparison analysis (Table 1) indicated that the differences in the mean strength values of 60/40 DC/ and 50/50 DC/BG composites compared to control DC were statistically significant at $p < 0.001$ ($p = 0.000654$ and $p = 0.000846$, respectively), while the differences between the 70/30 DC/BG composite and the control DC were statistically significant at $p < 0.05$ ($p = 0.001427$). Furthermore, the mean flexural strength value of the 70/30 DC/BG composite
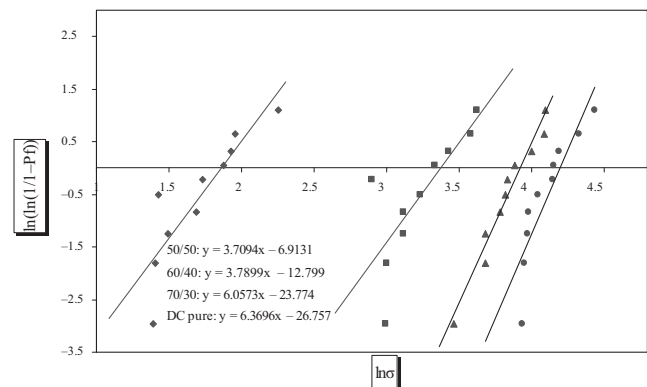
(48.5 MPa) was remarkably close to the value of 50 MPa that is suggested for the commercial use of a dental ceramic.

All 50/50 DC/BG specimens yielded mean strength values between 4.0 and 9.5 MPa, which were significantly lower ($p < 0.001$) compared to all other tested materials, and remarkably away from an acceptable value for good mechanical performance.

Computer generated Weibull plots of fracture stress are illustrated in Figure 4. The data points are described by a straight line produced by a least-squares fit (maximum likelihood method) of the fracture stress data. The mean m values are listed in Table 2. The highest m value was found for 70/30 DC/BG composite (6.06), while 50/50 had the lowest one (3.71). Most of the Weibull moduli calculated from the fracture results were in the range of 3.5–6.5. This range agrees with the m values commonly quoted for dental veneer ceramics [8].

Specimens groups		Mean Difference	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
50/50	60/40	−21.00753*	3.32161	0.000154	−30.2814	−11.7337
	70/30	−42.53678*	3.32161	0.000342	−51.8106	−33.2629
	DC (control)	−56.09249*	3.32161	0.000654	−65.3663	−46.8187
60/40	50/50	21.00753*	3.32161	0.000154	11.7337	30.2814
	70/30	−21.52925*	3.32161	0.000952	−30.8031	−12.2554
	DC (control)	−35.08496*	3.32161	0.000846	−44.3588	−25.8111
70/30	50/50	42.53678*	3.32161	0.000342	33.2629	51.8106
	60/40	21.52925*	3.32161	0.000952	12.2554	30.8031
	DC (control)	−13.55571*	3.32161	0.001427	−22.8295	−4.2819
DC (control)	50/50	56.09249*	3.32161	0.000654	46.8187	65.3663
	60/40	35.08496*	3.32161	0.000846	25.8111	44.3588
	70/30	13.55571*	3.32161	0.001427	4.2819	22.8295

* The mean difference is significant at the 0.05 level.

Table 1: One way analysis of variance of fracture toughness with associated Bonferroni's multiple comparison tests.

DC/BG	m
50/50	3.7094
60/40	3.7899
70/30	6.0573
DC control	6.3696

Table 2: Weibull's moduli.

4 Conclusions

Both FTIR spectra and XRD patterns of all powder samples confirmed the presence of both constituents in the composites. A gradual appearance of the bands that are attributed to leucite—which is the dominant phase in dental porcelains—, was recorded, while bands attributed to hydroxyapatite were further detected. Flexural strength values were in the range of 5–50 MPa and in combination to the Weibull's moduli of all DC/BG composites followed a linear relationship related to the increased amount of the dental ceramic in the composites. In conclusion, dental ceramics/bioactive glass composites with a high amount of dental ceramic can exhibit both mechanical integrity and bioactivity [2, 4] and consequently, can be potentially used in restorative dentistry.

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