Effect of Test Environment and Microstructure on the Flexural Strength of Dental Porcelains

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Abstract

Purpose: The objective of this study was to verify the influence of test environment on the flexural strength of dental porcelains with distinct microstructures.
Material and Methods: Disk-shaped specimens from three dental porcelains with distinct leucite content (VM: zero; CE: 12; NS: 22 vol%) were manufactured and tested for biaxial flexural strength in air and immersed in artificial saliva. The results were analyzed by means of two-way ANOVA and Tukey's test ($\alpha = 0.05$).
Results: The flexural strength (MPa) obtained for ambient air and artificial saliva environments, respectively, were: 110.0 ± 16.0 and 81.5 ± 10.8 for VM; 51.9 ± 4.0 and 42.0 ± 4.7 for CE; 72.0 ± 11.5 and 63.6 ± 5.8 for NS. A numerical decrease in the mean flexural strength was observed for all groups when specimens were tested under artificial saliva; however, the difference was only statistically significant for VM.
Conclusions: The results indicate that the effect of water immersion on the flexural strength of dental porcelains varies according to their leucite content, as only the material without leucite in its microstructure (VM) showed significant strength degradation when tested under water.

Leucite-reinforced dental porcelains are composed of a glassy matrix containing leucite particles, which were primarily introduced in this material for increasing its coefficient of thermal expansion (CTE) to make it compatible with the CTE of metallic frameworks.[1] Nonetheless, the addition of leucite crystals is also responsible for improving porcelain's fracture toughness[2] and Weibull modulus[3] due to toughening mechanisms like crack deflection. Dental porcelains have been widely used in restorative dentistry due to their relatively high hardness, good color stability, excellent biocompatibility, excellent capacity to mimic dental structure, and high chemical stability; however, these materials have some disadvantages, including low fracture toughness compared to metal and high susceptibility to water-assisted stress corrosion.[4]

The longevity of dental porcelain restorations is affected by the presence of volumetric flaws created during processing (microcracks, pores, inclusions, clusters) or superficial flaws caused by grinding or usage (surface voids, pits, scratches, cracks).[3] The stable extension of these preexisting defects under a stress intensity factor below the critical level is responsible for strength degradation over time. This phenomenon, known as slow crack growth (SCG), is even more critical under wet conditions.[5] The oral environment has several components (e.g., water from saliva, masticatory stresses, temperature and pH variations[6,7]) favoring the occurrence of SCG and eventually decreasing the material's lifetime.

To verify the effect of moisture on the performance of dental ceramics, some studies assessed the changes in the mechanical properties of these materials after storage in dry and wet conditions. Some of these works have tested the hypothesis that the porcelain's fracture toughness (K_{Ic}) would not change due to the presence of moisture, since K_{Ic} is a material constant for most ceramics and therefore should be independent of testing environment. In fact, a recent study[8] showed that the fracture toughness values obtained by means of fractrographic analysis for an aluminous porcelain (Vitadur Alpha, Vita, Bad Sackingen, Germany) tested in oil was statistically similar to values obtained in water. In addition, another study showed that after storing porcelains with and without leucite for 10 days in artificial saliva with different pHs (3.5, 7.0, 10.0), the fracture toughness was not negatively affected compared to the group kept in air for the same period of time.[6] In accordance with this, Drummond et al[9] showed that even though the fracture toughness values of several porcelains measured under water were lower than those obtained in ambient air, these differences between the experimental groups were not statistically significant.

Despite the above-cited evidence showing critical stress intensity factor is not affected by the test environment, Scherrer et al[4] demonstrated that the storage of a low-fusing dental glass (Duceram LFC) in deionized water (80°C) for 8 weeks resulted in a significant increase in the fracture toughness values obtained by two different techniques (indentation fracture, indentation strength). This result needs to be carefully analyzed because the low-fusing dental glass used in the study is regarded as an unusual material that undergoes crack tip blunting upon exposure to an aqueous environment.[10] It has been demonstrated that Duceram LFC was developed by incorporation of hydroxyl ion groups in the glass network, resulting in a material with a high CTE in the absence of a crystalline phase.[11] When this material reacts with the water from saliva, it forms a hydrolyzed surface layer more ductile than the bulk glass, which may be responsible for the observed increase in fracture toughness when stored in water. Other works have investigated the effect of aging in water on the flexural strength of this specific material with conflicting results showing either increase, decrease, or no effect of water exposure on flexural strength.[4,12-14]

The effect of the test environment on the flexural strength of various dental porcelains has also been investigated in previous studies. Depending on the porcelain tested, it has been demonstrated that compared to a dry condition, immersion in water is responsible for a decrease from 6 to 17% in flexural strength.[9] This deleterious effect of humidity on SCG was also reported in a study that estimated the stress corrosion susceptibility coefficient (n) using the indentation-fracture method.[1] This work showed that most porcelains tested showed a decrease in their n values when stored in artificial saliva for 6 months, indicating a higher susceptibility to SCG in this condition.[1]

Since environmental conditions play a fundamental role in the lifetime of dental porcelains, the objective of this study was to verify the influence of humidity on the fracture strength of dental porcelains with distinct microstructures. The hypothesis to be tested is that all materials will be negatively affected by immersion under water during testing.

Material and methods

The three dental porcelains used in this study (Table 1) were chosen to provide a systematic difference in leucite content.

Table 1 Materials used

Porcelain	Brand name/Manufacturer	Description
VM	Vita VM7/Vita, Bad Sackingen, Germany	Vitreous porcelain without crystal reinforcement indicated as veneering material for glass-infiltrated alumina cores
NS	Noritake Super Porcelain EX-3/Noritake-Kizai Co., Nagoya, Japan	Crystalline-reinforced porcelain used for porcelain-fused-to-metal or all-ceramic restorations
CE	Ceramco I/Dentsply Ceramco R and D, Burlington, NJ	High-fusing, leucite-based porcelain, used for metal-ceramic or all-ceramic restorations, containing dendritic leucite particles

Twenty disk-shaped specimens of each material were fabricated by mixing 1 g of porcelain powder with 0.4 ml of deionized water to form a slurry poured into a metal mold (15-mm diameter, 3-mm thick). Specimens were vacuum-sintered in a porcelain furnace (Keramat I, Knebel, Porto Alegre, Brazil) according to the manufacturer's recommendation. After sintering, specimens were machined in a surface-grinding device (MSG-600, Mitutoyo, São Paulo, Brazil) following the guidelines in ASTM C 1161[15] to reduce thickness to 1.3 mm and to obtain parallel surfaces. Then, one surface of each specimen was polished (Ecomet 2, Buehler, Lake Bluff, IL) to a final thickness of 1.00 ± 0.10 mm and a 1 μm suface finish (Extec, Buehler).

Microstructural analysis was performed under scanning electron microscope (SEM, Jeol Inc., Peabody, MA) after etching the polished surfaces of porcelains with 2% hydrofluoric acid solution (HF) for 15 seconds (for NS and CE) and 1 minute (for VM) to reveal the microstructure. The second-phase particle content, in volume percent (vol%), was obtained using ten SEM micrographs in image-processing programs (Adobe Photoshop 7.0, Adobe, Seattle, WA; ImageJ, NIH, Bethesda, MD). X-ray diffraction (XRD) was performed on the raw powders of CE and NS in a diffractometer (Rigaku Rint 2000, Tokyo, Japan) using Cu Kα radiation and a scan rate of 1° of 2θ per minute.

Ten specimens of each porcelain were tested for biaxial flexural strength (σ_F) using the piston-on-three-ball method[16] in different environments: (1) ambient air (21°C, relative humidity, RC = 40%), and (2) wet condition (immersed in artificial saliva[17] heated and maintained at 37°C) in a universal testing machine (Syntech 5G, MTS, São Paulo, Brazil) with a 5 mm/min crosshead speed. The value of σ_F was calculated according to:

$$\sigma_F = \frac{3F(1+\upsilon)}{4\pi t^2}\left[1 + 2\ln\frac{R_a}{b} + \frac{1-\upsilon}{1+\upsilon}\left(1-\frac{b^2}{2R_a^2}\right)\frac{R_a^2}{R^2}\right] \quad (1)$$

where F is fracture load, t is disk thickness, υ is Poisson's ratio, b is the radius of the loading area (piston radius, 0.85 mm), R is disk specimen radius, and R_a is the support circle radius defined by the three balls (4.0 mm).[18] The values of υ and Young's modulus (E) were determined by the ultrasonic pulse-echo method,[19] using a 200 MHz ultrasonic pulser-receiver (5900 PR, Panametrics, Waltham, MA), 20 MHz longitudinal and shear transducers with a delay material, and a coupling paste applied between the specimen and transducer.[20] According to the normality test (Kolmogorov-Smirnov, $p > 0.05$), the strength data were normally distributed. Therefore, the flexural strength results were analyzed statistically by means of two-way ANOVA and Tukey's test ($\alpha = 0.05$).

Fractographic analysis of the fractured porcelain discs was carried out under a stereomicroscope (CCD, Olympus, Center Valley, PA) at 200× magnification. SEM examination was performed on selected specimens to record representative fracture surfaces.

Results

The values obtained for υ and E (GPa) and the estimated leucite content (vol%) are presented in Table 2. No significant

Table 2 Poisson's ratio (ν), Young's modulus (E), and leucite content obtained for the three porcelains (standard deviations are shown in parentheses

Porcelain	ν	E (GPa)	Leucite content (vol%)
VM	0.22 (0.01)^{a}	66.0 (0.9)^{a}	0^{c}
CE	0.20 (0.01)^{a}	69.7 (0.7)^{a}	12 (1.0)^{b}
NS	0.21 (0.01)^{a}	67.7 (0.9)^{a}	22 (2.0)^{a}

$^{a-c}$Different superscripts indicate statistically significant difference in columns, $p < 0.05$.

differences were observed in these constants for the materials studied ($p < 0.05$). The microstructures of the porcelains studied are shown in Figure 1. Porcelain VM did not show second-phase particles after etching with HF; however, it was possible to note regions with different corrosion rates, probably related to the starting glass powder (Fig 1A). For CE and NS (Figs 1B,C) it was possible to note second-phase particles heterogeneously distributed in the glassy matrix. The peculiar dendritic structure of the leucite particles of porcelain NS is also shown in Figure 1D. The leucite particles in porcelain CE had an equiaxial shape (Fig 1B). XRD analysis of CE and NS detected a large band (representative of the amorphous phase) and diffraction peaks corresponding to the crystalline leucite phase ($KAlSi_2O_6$) according to JCPDS card no. 38–1423.

For strength data, two-way ANOVA showed that both main factors (material and environment) were statistically significant ($p < 0.05$); however, the interaction of these factors was also statistically significant ($p < 0.05$),indicating that the effect of the environment on the flexural strength depended on the type of material tested. In fact, though there was a numerical decrease in the mean flexural strength for all materials when specimens were tested under artificial saliva, the difference between strength values was only statistically significant for porcelain VM (Table 3). For the two non-statistically significant pairwise comparisons (CE-air $*$ CE-saliva; NS-air $*$ NS-saliva, Table 3), the statistical powers was calculated based on the difference between means, sample size, standard deviations, and α of 0.05, resulting in power of 0.998 and 0.603, respectively.

Table 3 Mean biaxial flexural strength ± standard deviation and variation observed from porcelains studied as a function of test environment (ANOVA and Tukey's test, $\alpha = 0.05$)

	Biaxial flexural strength (MPa)		
Test condition	VM	CE	NS
Ambient air	110. ± 16^{a}	51.9 ± 4.0^{de}	72.0 ± 11.5^{bc}
Artificial saliva	81.5 ± 10.8^{b}	42.0 ± 4.7^{e}	63.6 ± 5.8^{cd}
Variation (%)	−25	−19	−12

$^{a-e}$Different superscripts indicate statistically significant difference in columns, $p < 0.05$.

The fractographic analysis of the broken disks showed that for all experimental groups, the critical flaw was located at the tensile surface of the specimen and had an approximate

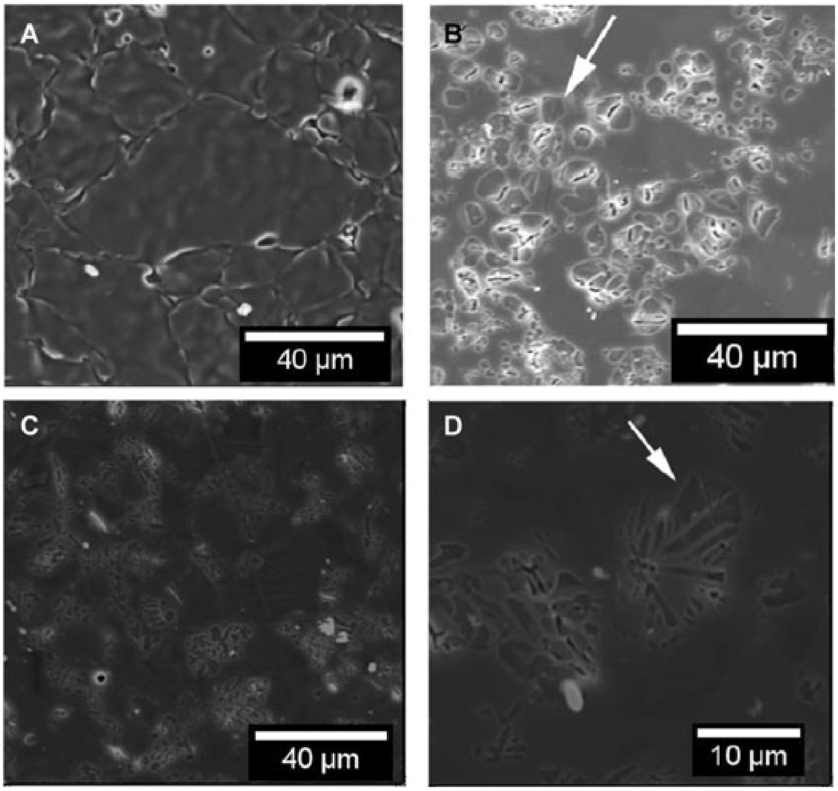

Figure 1 Micrographs of the polished surface of porcelains VM (A), CE (B), and NS (C and D). The arrows indicate the equiaxial leucite particles in (B) and dendritic leucite.

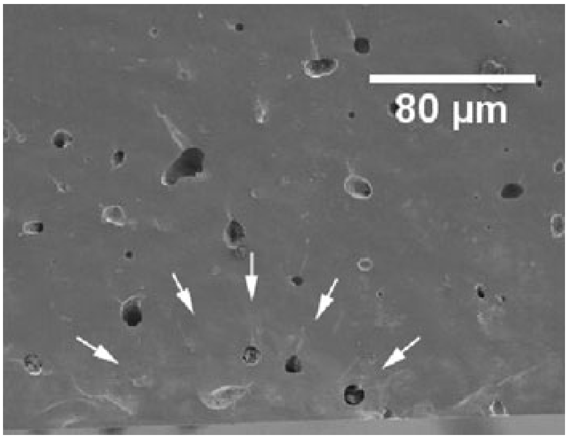

Figure 2 Representative fracture surface of porcelain VM7 fractured in ambient air. The arrows mark the boundary of the critical flaw.

semi-elliptical shape (Fig 2). Rougher fracture surfaces were observed in porcelains CE and NS probably due to crack deflection around the leucite particles and clusters. Porcelain VM showed a much smoother fracture surface, facilitating the identification of the critical flaw. A thorough fractographic analysis of porcelains CE and VM (as well as other leucite-based porcelains) has been carried out in previous studies by our research group and can be found elsewhere.[2,5,6,21]

Discussion

The hypothesis of this study was partially accepted since only porcelain VM experienced a significant decrease in its flexural strength under moist condition, as compared to ambient air. This is a result of the chemical interaction between the superficial flaws of the porcelain and the reactive environment, such as water or water vapor.[22] This mechanism may be described as the hydrolysis of covalent metal-oxide-metal ceramic bonds (M-O-M) in preexisting cracks under tensile stress. Once the M-O-M bond is broken, forming two hydroxides (M-OH), (Fig 3), the crack grows at low velocities until reaching its critical length at a given applied stress, leading to catastrophic failure.[23]

This study showed that the strengths of both leucite-based porcelains (CE and NS) was not significantly affected by immersion in water. This finding is most likely related to the presence of second-phase particles in these materials, which are known to influence the material's mechanical behavior in distinct manners. It has already been demonstrated that second-phase particles are responsible for an increase in the fracture toughness[2] and in the stress corrosion coefficient, n (the higher the n value, the lower susceptibility of the material to the SCG phenomenon).[6] Such improvements in mechanical properties may be explained by the crack deflection mechanism that occurs when a crack changes its propagation direction after meeting a second-phase particle.[2,24] This crack interaction with the material's microstructure often occurs when the crack comes upon tangential compressive stresses, and is then guided around the particles or clusters by radial tensile stresses, changing its propagation path and reducing the stress intensity factor at the crack tip.[2]

Another possible reason for the absence of a significant effect of water immersion for the particle-reinforced materials lies in the idea that the presence of leucite particles reduces the total amount of glassy matrix exposed to the reactive environment, reducing the area of amorphous glass subjected to stress corrosion. This could also help explain the significant decrease in flexural strength observed for porcelain VM, as this material is entirely vitreous.

The large mismatch between the CTE of the second-phase particles and the porcelain's glassy matrix gives rise to stresses when the porcelain is cooled after sintering. This causes microcracks to appear in and around the crystal clusters, which may separate or decouple the particle from the surrounding matrix.[2] In fact, a work by Borom[25] evaluated the results from the literature and showed that particles introduced in a ceramic matrix do not limit flaw sizes, but may produce surface flaws in proportion to particle diameter. In addition, Borom stated that the presence of particles with high elastic modulus, coherently bonded in a brittle matrix increases the load to fracture in proportion to the increase in the system modulus; however, the expected strength enhancement may not be achieved because

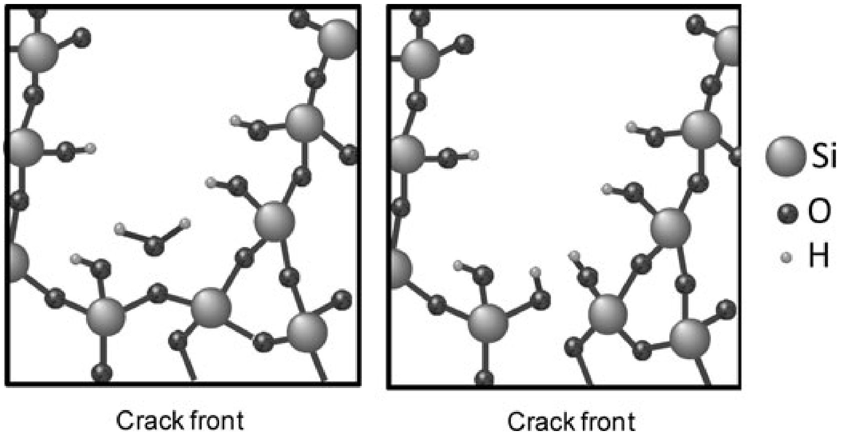

Figure 3 Schematic molecular images showing the hydrolysis of covalent metal-oxide ceramic bonds under tensile stress leading to SCG.

of particle-induced flaws. Since porcelain VM is particle-free, the absence of flaws in its microstructure may have contributed to the significantly higher flexural strength values observed for this material, irrespective of the test environment (Table 3).

From the clinical standpoint, the results of the present study showed that the porcelain chosen to construct a dental prosthesis is likely to affect its clinical outcome. The porcelains tested are indicated for veneering different types of frameworks. In this way, CE and NS should be used over metallic frameworks, and VM is suited for ceramic frameworks; however, these materials can also be used to construct all-ceramic inlays, onlays, and laminate veneers. In this case, one should choose a material with a mechanical strength not significantly affected by immersion in water, such as porcelains CE and NS.

Some limitations of the present study should be pointed out, as the experimental design used in this work is different from what is found in the clinical situation. In the oral cavity, the breaking stresses are usually lower due to fatigue and cyclic loading. Moreover, the surface finishing of the disk specimens is different from that found in dental restorations, since the disks are mirror-polished in a polishing machine, and the real restorations are usually glazed.

Conclusion

Within the limitations of this study, the results indicate that the effect of water immersion on the flexural strength of dental porcelains varies according to the porcelains' leucite content, as only the material without leucite in its microstructure (VM) showed significant strength degradation when tested under water.

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