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Water as Veneering Modeling Liquid Affects Microhardness of Glassy Matrix Veneering Ceramic

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ABSTRACT

Purpose/Aim: To evaluate the effect of different veneering liquids used for modeling on microhardness, fracture toughness and biaxial flexural strength of a glass-veneering ceramic. Material and methods: The manufacturer recommended modeling liquid (ML), distilled water (DW), isopropyl alcohol (IA), 0.5% (P05), 1% (P1), and 2% (P2) polyethylene glycol solutions were mixed with feldspathic ceramic powder to form disc-shaped samples (n=20, 15 mm × 1.2 mm). After sintering, samples were mirror-polished and subjected to Vickers indentation (n=5) for measurement of microhardness and fracture toughness. The remaining 15 samples from each group were subjected to biaxial flexural strength. Data were subjected to one-way ANOVA and Weibull analysis. Results: The microhardness was affected by veneering liquid (p=0.002): DW promoted higher microhardness values than ML and IA. Fracture toughness (p=0.301) and flexural strength (p=0.930) were not affected by the veneering liquid but Weibull parameters were affected. All groups presented surface pores under high magnification. Conclusion: Even though the use of DW led to higher values of surface microhardness than the ML, all obtained values are inside the range of enamel microhardness values reported in the literature. Such parameters may affect antagonist wear and should be reported in clinical trials.

INTRODUCTION

The clinical chipping of veneering ceramic on zirconia-based restorations is continuously reported in the literature. It is the main failure for tooth and implant-supported fixed dental prosthesis (FDP),[1] reaching 20% of the restorations after 5 years.[2] Porosities in veneering ceramic,[3] residual thermal stresses,[4] inadequate support provided by framework[5] and even the low strength of glass-veneering ceramics compared to other veneering ceramics[6] are the main reasons for ceramic fracture. However, veneering glass-ceramic over a ceramic framework is still considered a more esthetic approach than monolithic restorations.[7]

The veneering process, in turn, is very sensitive and dependent upon skilled operators. One of the reasons for that is the difficulty in using a correct powder/liquid ratio - with the best results (highest apparent density and the lowest total porosity) reported for the ratio 2.64 g/mL.[8] The type of liquid used for condensation of veneering ceramic also seems to affect the properties such as density, Vickers hardness and flexural strength,[9,10] but scientific evidence is scarce on this topic.

The veneering liquid creates the pellicle necessary to gather the particles together by moving the powder particles closer. The sintering process will then promote the fusion of the ceramic powder, leading crystals to an unorganized liquid state; the subsequent cooling originates a crystalline solid followed by contraction of the material due to the atomic packing and normal cooling contraction.[11]

Water is a universal solvent commonly used by technicians. The presence of water decreases the viscosity of the veneering ceramic, decreasing the glass crystallization rate.[12] The water present in the powder-liquid mixture before crystallization creates a static hydroxyl group,[13] and ceramic becomes more brittle. Moreover, the presence of water in veneered zirconia restorations has been related to the fretting of zirconia grains at the interface between the ceramics.[9] Isopropyl alcohol is another solvent type that would result in low water content in the slurry and a high strength of the final sintered glass-veneering ceramic. One possibility is polyethylene glycol - a flexible and hydrophilic polymer molecule,[14] used as a lubricating coating for different surfaces in the presence or absence of water[15] which can create very high osmotic pressures.

The layering of metal or ceramic frameworks is handwork, and technical personal preferences are applied by ceramists, modifying the manufacturers' instruction during the fabrication of the restoration. Thus, the aim of this study was to evaluate the hardness, fracture toughness and strength of sintered feldspathic ceramic prepared by mixing the powder with different liquids. The tested hypothesis is that the manufacturer's recommended veneering liquid will provide the best mechanical properties to the final sintered glass-ceramic.

MATERIAL AND METHODS

SPECIMEN FABRICATION

Veneer ceramic disc-shaped samples were fabricated from feldspathic ceramic powder (dentin, VITA VM9, VITA Zahnfabrik, Bad Säckingen, Germany) mixed to one of the following liquids (n = 20): VITA modeling liquid (ML, VITA Zahnfabrik, Bad Säckingen, Germany); distilled water (DW), isopropyl alcohol (IA), 0.5% polyethylene glycol solution (P05), 1% polyethylene glycol solution (P1), 2% polyethylene glycol solution (P2). The ceramic powder was weighted (0.650 g) and mixed to 2 µl of the liquid. A homogeneous slurry was obtained and inserted in one increment into a metallic matrix (18 mm x 1.5 mm). The slurry was condensed by a cylindrical metallic piston (18 mm in diameter, flat tip), and absorbent paper was used to remove the liquid excess.

Discs were removed from the matrix and sintered in a specific furnace (VACUMAT 40, VITA Zahnfabrik, Bad Säckingen, Germany) according to manufacturer's instructions (dentin 1st firing; 910 ºC/1 min). After sintering, discs were inspected for cracks and replaced if needed. Then, samples were mirror-polished (ECOMET/AUTOMET 250, Buehler, Lake Buff, Ill, USA – sandpaper of 400, 600, 1200 and 2000 grit), and inspected for cracks once more. The final dimension of the discs was 12 mm x 1.2 mm (ISO 6872).

MICROHARDNESS TEST

Five samples from each group (n=5) were used for measuring the microhardness of the ceramic. Five Vickers indentations (49 N/ 20 s) were performed in each sample (Shimadzu MicroHardness Tester HMV G20, Shimadzu Corporation, Duisburg, Germany). One indentation was performed in each quadrant, and one in the center of the disc. The distances between both diagonals of the indentation were measured and the hardness value was calculated according to Equation 1.[16]

$$H = 0.5\frac{P}{a^2}$$

where "H" is the hardness, "P" is the applied load, and "a" is the average indentation half-diagonal. The mean of the 5 values obtained was considered the hardness value of that sample. Obtained data were subjected to Kolmogorov-Smirnov test for data distribution and then, subjected to one-way analysis of variance, followed by Tukey *post hoc* test (α=0.05).

FRACTURE TOUGHNESS

The same samples used for the microhardness test were also used for fracture toughness measurement (n=5). After Vickers indentation, the sample was observed regarding the criteria (1) cracks should emanate from the corner of the indentation, (2) presence of only four radial cracks, (3) no chipping and (4) cracks without branching.[17] The cracks emanating from the corners of each indentation were measured under 400 × magnification. The distance between the center of the indentation and the end of the crack (c), the elastic modulus (E - 66.5 GPa) and the Vickers hardness (H - measured as previously mentioned) were used for the calculation of the fracture toughness (K_{Ic}) value (Equation 2).[18]

$$K_{Ic} = 0.016\left(\frac{E}{H}\right)^{0.5}\left(\frac{P}{c^{1.5}}\right)$$

Obtained data were subjected to Kolmogorov-Smirnov test for data distribution and then, subjected to One-way analysis of variance (α=0.05).

BIAXIAL FLEXURAL STRENGTH TEST

Samples (n=15) were tested according to ISO 6872, on the piston-on-three balls assembly. Discs were positioned on the support balls (Ø = 3.2 mm). A metallic cylindrical piston with a flat tip (Ø 1.6 mm) applied an increasing load (1 mm/min, EMIC DL 3000, EMIC, São José dos Pinhais, Brazil) until fracture of the sample. The maximum applied load was recorded and the strength was calculated according to equation 3.

$$\sigma = -0{,}2387 \times \frac{p(X-Y)}{b^2}$$

where "σ" is the flexural strength (MPa), "p" is the maximum applied load (N), "b" is the sample thickness at the fracture origin (mm) and X and Y where determined according to equations 4 and 5.

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$$X = (1+\vartheta)ln\left(\frac{r_2}{r_3}\right)^2 + \left[\frac{(1-\vartheta)}{2}\right]\left(\frac{r_2}{r_3}\right)$$

$$Y = (1+\vartheta)\left[1 + ln\left(\frac{r_2}{r_3}\right)^2\right] + (1-\vartheta)\left(\frac{r_1}{r_3}\right)$$

where "ϑ" is the Poison's ratio (0.23), "r_1" is the radius of support cycle formed by the three support balls (5 mm), "r_2" is the radius of the loaded area (0.8 mm) and "r_3" is the radius of the sample (mm).

Obtained data were subjected to Kolmogorov-Smirnov test for data distribution and then, subjected to one-way analysis of variance ($\alpha = 0.05$). Strength data were also subjected to Weibull analysis (DIN E ENV 843-5) for determination of the modulus (*m* – reliability of the material) and the characteristic strength (σ_0 - strength at a failure probability of 63%).

Samples were gold-sputter coated and images from samples' surfaces (n=1) were performed by a scanning electron microscope equipped with a field emission gun (SEM-FEG) in 1000× and 5000× magnification for observation of surface pores.

RESULTS

Microhardness was affected by the veneering liquid used (p=0.002): the DW promoted higher microhardness values than ML and IA *(Table 1)*. Fracture toughness was not affected by the veneering liquid (p=0.301) *(Table 1)*.

Table 1. Microhardness and fracture toughness according to veneering liquid.

Veneering liquid	Mean hardness (GPa)*	Mean fracture toughness (MPa m$^{1/2}$)
ML	3,07 (0,84)^{bc}	3.33 (0.71)
DW	4,08 (0,35)^{a}	3.59 (0.32)
IA	2,48 (0,11)^{c}	2.70 (0.73)
P05	3,44 (0,33)^{abc}	2.96 (0.34)
P1	3,60 (0,26)^{ab}	3.16 (0.38)
P2	3,28 (0,36)^{abc}	3.11 (0.51)

*Similar letters indicate statistical similarity.

The biaxial flexural strength was also not affected by the veneering liquid used (p=0.930) *(Table 2)*. The reliability and characteristic strength were similar between all groups.

After the strength test, samples presented 2 to 5 fragments, that were recorded and correlated to the individual strength of each sample *(Figure 1)*: the tendency is that the number of fragments enhances according to the increase of the flexural strength of the sample.

Table 2. Flexural strength and Weibull parameters according to veneering liquid.

Veneering liquid	Mean Flexural strength (MPa)	*m* (IC)*	σ_0 (IC)*
ML	61.45 (10.78)	6.56 (3.79 - 9.16)	65.89 (59.64 - 72.76)
DW	60.05 (14.33)	4.97 (2.87 - 6.94)	65.41 (57.35 - 74.56)
IA	65.75 (15.89)	5.21 (3.01 - 7.28)	67.42 (59.49 - 76.39)
P05	62.09 (14.07)	4.61 (2.66 - 6.44)	72.00 (62.49 - 82.92)
P1	60.30 (18.81)	3.25 (1.88 - 4.54)	67.72 (55.39 - 82.74)
P2	62.27 (17.67)	4.14 (2.26 - 5.88)	68.62 (57.74 - 81.65)

*Statistical difference is determined by the absence of overlapping between confidence intervals

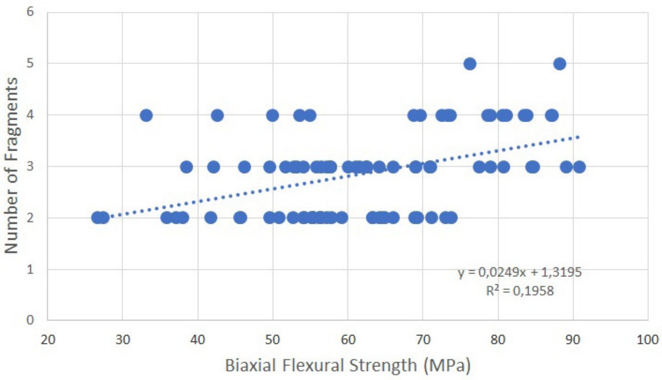

Figure 1: Number of fragments by the flexural strength of each sample, despite veneering liquid.

Images from SEM-FEG are presented in figure 2. The surface pattern is similar to all veneering liquids, with some pores in the glassy matrix.

DISCUSSION

The alternation in veneering liquid for manufacturing of feldspathic veneering ceramic promoted alterations in the hardness of the material, but did not affect the fracture toughness and strength of the ceramic. The use of water and polyethylene solutions as veneering liquids promoted the highest hardness values. The study hypothesis was rejected.

Potassium aluminosilicate glasses, as the case of feldspathic ceramic, are hygroscopic and absorb moisture at room temperature.[19] The water promotes more easily and

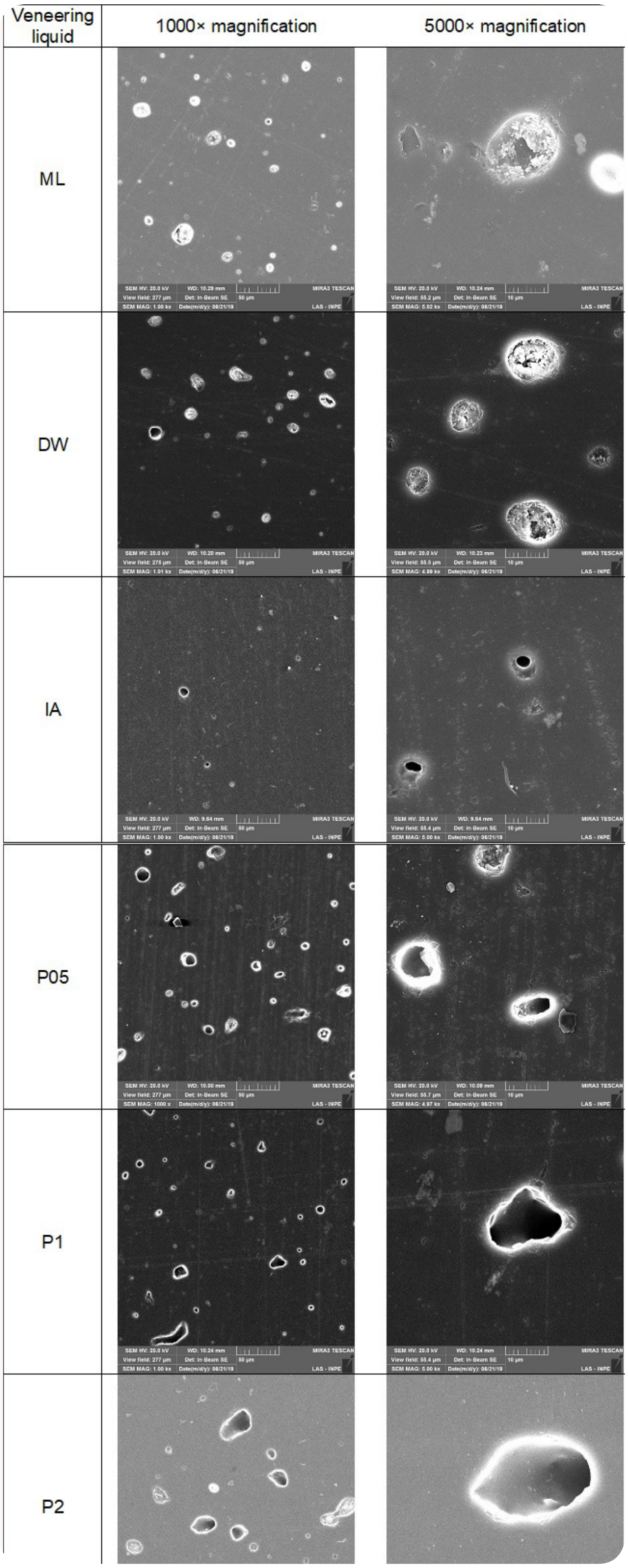

Figure 2: SEM-FEG images of samples surfaces showing pores in the glassy matrix of ceramic: VITA modeling liquid (ML), distilled water (DW), isopropyl alcohol (IA), 0.5% polyethylene glycol solution (P05), 1% polyethylene glycol solution (P1), 2% polyethylene glycol solution (P2), with magnifications of 1000x and 5000x.

rapidly crystallization.[19] This water-enhanced crystallization increases nucleation and crystal growth.[20,21] The water present during mixing and veneering of glass-ceramic promotes the formation of a hydroxyl group (HO),[13] which is static, decreasing the ability of the material to suffer plastic deformation, resulting in higher surface hardness values *(Table 1)*.

The hardness, together with surface roughness, coefficient of friction, crystal size and elastic modulus of ceramic, are properties associated with the wear of the material and the opposed substrate.[22] The wear of the enamel opposite to a ceramic restoration is not desirable, as the wear of the ceramic itself: in a recent study, the clinical fractures found in the veneering ceramic of zirconia and metal restorations had their origin located at wear facets on the occlusal surface of the restoration.[23] The ideal material would have the physical and mechanical properties similar to that of tooth tissues. Since the reported hardness of enamel is between 3.70 GPa[22] and 4.12 GPa.[24] Despite the increase in hardness presented by groups DL, P05, P1, P2, compared to ML, the produced ceramics presented hardness similar to enamel *(Table 1)*.

The fracture toughness was not affected by the veneering liquid used for ceramic fabrication *(Table 1)*. This property represents the ability of the material to resist crack propagation, and may be positively related to the volume and size of ceramic crystals.[25,26] The amount of water in the pre-sintered ceramic samples also did not affect fracture toughness of glass-ceramic in a previous study.[27]

The indentation fracture method was used for measuring the fracture toughness of the ceramic. This test was applied in previous similar studies,[10,28] since provides the opportunity of rapid evaluation in small samples, is convenient, and maintain the popularity for evaluating properties in ceramic materials. It is considered less accurate than the tests performed by macroscopic tests. But the values in the present study varied between 11 and 27%, presenting normal distribution in data analysis.

The flexural strength of the feldspathic ceramic was not affected by veneering liquid *(Table 2)*. The remove of water excess in leucite ceramics improved its flexural strength,[27] and increase in the crack initiation stress,[29] but authors affirm that the mechanism for strength improvement is not clear.[27] The microstructure of glass-ceramics – crystal size, crystal volume – influence the mechanical properties of the material,[28] but previous studies showed by XDR and SEM investigations that DW or ML did not affect the resultant final microstructure.[9] The fracture pattern regarding flexural strength *(Figure 1)* is in accordance with the literature.[30]

The patter of porosities presented by each veneering liquid in the ceramic surface was similar *(Figure 2)*, corroborating with the similarity found between groups regarding fracture toughness and flexural strength. In the present study, the veneering liquid was measured by its volume for mixing with porcelain powder. Previous studies used the weight of the liquid, which may result in different liquid volumes according to liquid density.[9] Higher volume of liquid, higher wetting of powder, resulting in less porosity of the samples and improved mechanical properties.[9] Despite no alteration was promoted by different veneering liquids in the strength of ceramic, the longevity of materials which are fabricated with variations in technique should be investigated associated to aging and fatigue tests.

This study evaluated the veneering ceramic fabricated isolated. Other condition such the layering of metal or other ceramics may present different results. Moreover, no ageing simulation was performed, which is an important factor in the clinical behavior of dental ceramics.

CONCLUSION

The veneering liquid did not affect the fracture toughness and flexural strength of ceramic but Weibull parameters were affected. Even though the use of distilled water led to higher values of surface microhardness than the modeling liquid indicated by the manufacturer, all obtained values are inside the range of enamel microhardness values reported in the literature.

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