The effect of slurry preparation methods on biaxial flexural strength of dental porcelain

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Statement of problem. One-step and incremental mixing procedures are currently used to produce dental ceramic pastes. In the ceramic industry, high quality is obtained using one-step mixing, but in dentistry, the best method has not been yet determined.

Purpose. The purpose of this study is to evaluate the effects of 2 mixing techniques on the biaxial flexural strength and microstructure of dental porcelain.

Material and methods. Feldspathic porcelain discs (2 x 15 mm in diameter) were produced and divided according to the ceramic paste preparation method, powder-liquid incremental mixing group (n=50) or one-step mixing, as a control group (n=50). Specimens were tested for biaxial flexural strength and characterized using porosimetry, relative humidity, SEM/EDS, XRD, and FT-IR analyses. Statistical analysis was conducted using Weibull statistics. The Weibull modulus, characteristic strength and relative humidity were compared between groups, using Student's t-test and Mann-Whitney U test (α=.05).

Results. The powder-liquid incremental mixing group showed significantly higher values (SD) of Weibull modulus (6.74 (0.70), P<.001) and characteristic strength (79.87 (2.01) MPa, P<.001) when compared to the one-step mixing group (4.94 (0.94) and 75.95 (2.61) MPa). Significantly lower mean (SD) relative humidity values (P=.009) were found for powder-liquid incremental mixing group (20% (0.5%)) compared to one-step mixing group (22% (1%)). XRD spectra showed that the one-step mixing group produced higher amounts of the amorphous phase.

Conclusions. Specimens produced by the incremental mixing technique showed higher biaxial flexural strength than one-step mixing. (J Prosthet Dent 2011;105:308-314)

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Clinical Implications

Ceramic slurry preparation may be a relevant factor in the performance of dental ceramics. A small variation in the relative humidity of green-state discs after de-molding, results in a decrease in Weibull modulus and characteristic strength for the one-step mixing method, which could be related to the presence of increased amounts of amorphous phase.

Dental feldspathic porcelains are composed of an amorphous matrix (K_2O-Al_2O_3-SiO_2 glasses) with dispersed leucite particles and oxides, responsible for lowering the melting temperature of the material and providing color and opacity.[1,2] Low-fusing porcelains in fixed dental prostheses (FDP) exhibit desirable properties such as excellent esthetics,[3] high biocompatibility,[4] high wear resistance,[5,6] and high chemical stability.[7] However, their brittle behavior can jeopardize long-term durability of FDP, which can result in catastrophic failures under physiological or pathological loading during oral function.[8-10]

The long-term durability of porcelain restorations may be increased using several strategies that might be classified as preventive, to avoid the formation and propagation of large and heterogeneously distributed flaws,[11-16] thus, having direct effect on the mechanical properties; and corrective, focused on restoration repair procedures after failure.[17-18]

Dental porcelain processing involves several stages that must be carefully controlled to guarantee the final quality of the prosthetic work. After industrial production of high quality powders with controlled particle size and complex chemical composition, other important steps are performed by both the dental technician and the dentist, including the mixing of ceramic paste, followed by fabrication of the prostheses, sintering and polishing.[14] Avoiding the introduction of defects during the manufacture of porcelain teeth is essential to obtain restorations with high mechanical properties, and therefore, longer clinical lifetimes.[19]

In the ceramic industry, optimized proportions are used to produce the porcelain slurry in which water is mixed with the porcelain powder in a highly controlled manner. In dentistry, porcelain slurries are also obtained by mixing water with the porcelain powder, however, an empirical approach is used in the laboratory to obtain a paste consistency that results in intra and inter operator variability.[15,20]

There are 2 different methods commonly used for preparing ceramic pastes in the dental laboratory, the incremental technique, in which water is added in small increments to the powder, and the one-step technique, in which a predetermined amount of water is added in a single step to the porcelain powder. Both techniques aim at obtaining a final creamy porcelain slurry with an optimal powder/liquid ratio of 2.8 g/mL.[15,19] When this optimal powder/liquid ratio is obtained, the liquid occupies small spaces between the ceramic particles and acts as a lubricant for particle movement, producing highly dense green-states and final fired-ceramics,[8,9,15,19-22] with homogeneously distributed microdefects and uniform porosity.[23-25] Excess water in the porcelain slurry is a problem, because excess water in the cast will evaporate and result in higher porosity. Moreover, excess water may react with some components of the porcelain and induce changes in the microstructure of the final fired product, changing the relative amounts of crystalline and glassy phases.[26-30]

Weibull statistics is a commonly used method for characterizing the failure of brittle materials under different flexural configurations.[13,31,32] The Weibull distribution is an extremely valuable distribution with descriptors such as Weibull modulus (a dimensionless dispersion measurement) and characteristic strength (stress value at which the probability of failure (Pf) is 63.2%). The Weibull distribution is homologous to the descriptors used in normal distribution such as standard deviation (a dispersion measurement) and mean (a stress value where the Pf is 50%) values.[32] The accuracy of the Weibull analysis for dental ceramics depends on the quality of the specimens tested, as well as proper selection of the test design and algorithms to estimate the descriptors based on the obtained data.[32] A high Weibull modulus is advantageous, because it indicates a low structural variability among specimens and a more predictable failure behavior.[33] A low structural variability of the fired ceramic restorations, at least theoretically, might reduce the incidence of fractures and chips, therefore reducing the need for repair and replacement of FPR.

The purpose of the study was to evaluate the biaxial flexural strength and microstructure of porcelain specimens obtained by 2 different slurry preparation methods (powder-liquid incremental and one-step mixtures). The hypothesis was that there would be significant differences between the Weibull parameters calculated from biaxial flexural strength of specimens obtained by the different preparation techniques.

MATERIAL AND METHODS

In this study low fusing porcelain (VITA Omega 900, Dentin, Color 4R

1.5, Vita Zahnfabrik, Bad Sackingen, Germany) was used to produce discs 15 mm in diameter and 2 mm thick. The specimens were divided in 2 groups, according to the preparation method.[33] Powder-liquid incremental group discs (n=50) were obtained by incremental addition of the liquid to the powder using a dental laboratory brush (#8; Smile Line, St-Imier, Switzerland). One-step group discs (n=50) were obtained by single addition of the liquid to the powder using 1.82 g of powder per 0.66 mL of distilled water.[15] All discs were fabricated using a 18 mm diameter plastic syringe (Becton Dickinson & Co, Franklin Lakes, NJ), which was sectioned and its plunger was polished and used as the disc base.[34]

In both methods, the ceramic paste was prepared at room temperature on a glass tile and then loaded into the syringe with a ceramic brush; the ceramic was condensed using an absorbent paper towel to remove the excess water.[34] The plunger of the syringe was placed 3 mm away from the top-edge using a periodontal probe. The surface of the disc was leveled against a flat surface to ensure a uniform thickness before removing the disc from the syringe.[15] The green-state specimens were removed from the syringe with a scalpel and placed on a refractory platform (Vita Zahnfabrik). All discs were heat-treated in an oven (Vacumat 40; Vita Zahnfabrik) according to the procedure described by the manufacturer. After sintering, the surfaces of the discs were polished with a series of abrasive papers (400, 600, and 1200 grit) using a polishing machine (LaboPol-5; Struers, Copenhagen, Denmark).[21]

Biaxial flexure strength was determined with a ball-on-ring design,[19,25] where each disc was supported on a steel ring (9.86 mm diameter). Load was applied centrally through a spherical ball indenter (5 mm diameter) in a universal test machine (650R; TestResources Inc, Shakopee, Minn) with a crosshead speed of 0.5 mm/min until failure. The maximum strength of the biaxial flexure test for each disc was calculated according to:[25]

$$\sigma_{max} = (P/h^2)\{(1+\nu[0.485x\ln(a/h) + 0.52] + 0.48\}$$

where σ_{max} is the maximum tensile strength (MPa), *P* is the measured load at fracture (N), h is the thickness of the disc (mm), ν is the Poisson's ratio (0.25 for porcelain),[19] and *a* is the radius of the supporting ring (mm).

The Weibull distribution was used to calculate fracture probabilities of each group as a function of applied stress. The equation used for the Weibull-parameter distribution function was:[35]

$$P_f = 1 - \exp\{- \int_S [(\sigma/\sigma_0)]^m dS\}$$

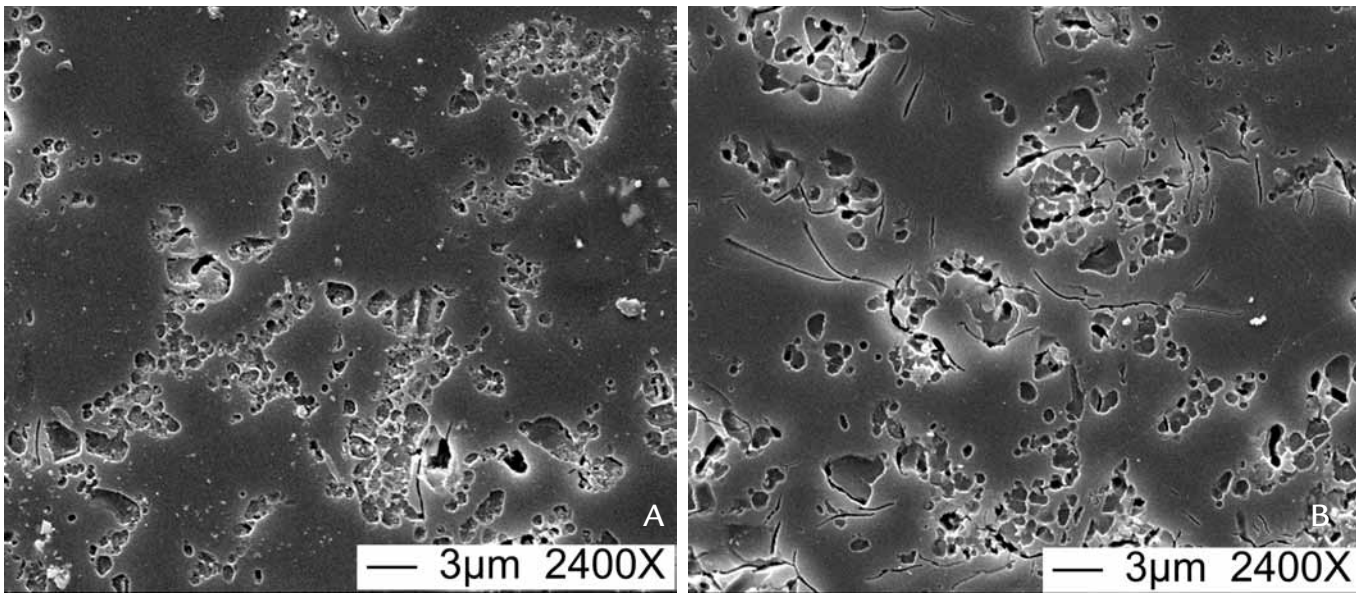
Where σ is stress; σ_0 is scale parameter or characteristic strength (stress level at which 63.2 % of the specimens failed); *S* is the surface under stress; and *m* is a dispersion parameter or Weibull modulus related to the slope of the distribution.

Specimens obtained by the different processing methods were characterized in terms of chemical composition, powder/liquid ratio, fractography, crystallinity and porosity. These analyses were performed for the commercial raw powder, green-state and sintered discs. To quantify the relative humidity after de-molding, 5 green-state discs per group were tested at 180°C using a thermal infrared moisture balance (Eurotherm, Gibertini Elettronica; Milano, Italy), which has an automatic function to determine the stable weight (DSW). The DSW value is defined when the variation in the weight is lower than 0.01 mg for 9 minutes.

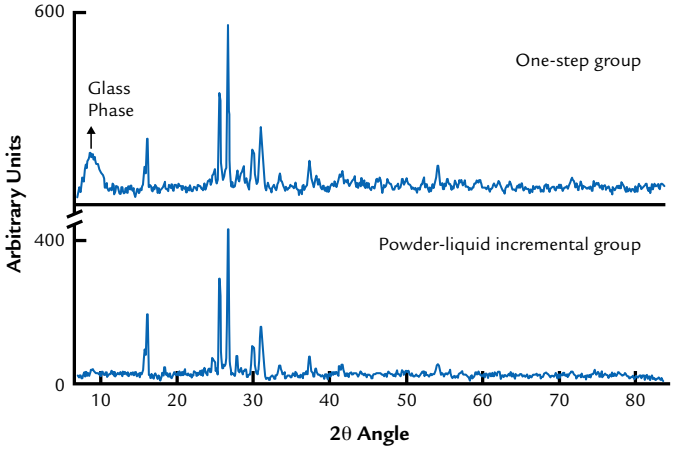
Compositional analysis of the raw powders was accomplished using x-ray fluorescence (PW2404 X-ray Spectrometer; PANalytical B.V., Almelo, The Netherlands) and atomic absorption spectroscopy (Solar 929 AA Spectrometer; ATI Unicam, Cambridge, UK). For Fourier transformed infrared (FT-IR) analysis, fractured discs were evaluated using a spectrometer (System 2000; PerkinElmer, Wellesley, Mass)[12,36,37] with an attenuated total reflectance accessory (Golden Gate Single Reflection Diamond ATR; Specac, Smyrna, Ga). For the FT-IR characteristic spectrum of both groups, 3 discs fractured under biaxial flexural test were selected from the tenth percentile where the fracture was obtained at lowest strain. In each disc, at least 3 spectra were obtained at different areas (each spectrum is the average of 100 scans between 400 cm^{-1} to 4000 cm^{-1} with 4cm^{-1} resolution). Baseline correction and mean spectrum were then calculated using numerical software (Matlab, Version R2008b; Mathworks Inc, Natick, Mass).

Two different types of surfaces, fractured and polished, (both etched with 2% HF hydrofluoric acid)[11] of 3 discs (within the tenth lowest percentile strength) of both groups were examined using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) microprobe (JEOL JSM-6301F, Tokyo, Japan). X-ray diffraction (XRD) was used to identify the crystalline phases and detect the presence of amorphous phases in specimens using the 2θ-angle range between 5 and 85 degrees with a scan speed of 0.02 degrees/s (X´Pert PRO alpha-1, PANalytical B.V., Almelo, The Netherlands), using a diffracted x-ray detector (RTMS X´Celerator; PANalytical B.V.) The XRD characteristic spectrum was obtained from the mean of 3 discs per group in the tenth lowest percentile, using dedicated software (High Score Plus XRD; Version 2.2, PANalytical B.V.). The detected peaks were matched using the crystallographic database of International Centre for Diffraction Data (ICDD) in the Powder Diffraction File (PDF, 2004).[14,38] Open porosity volume was calculated following the determination of the apparent density based on the Archimedes water displacement method.[39]

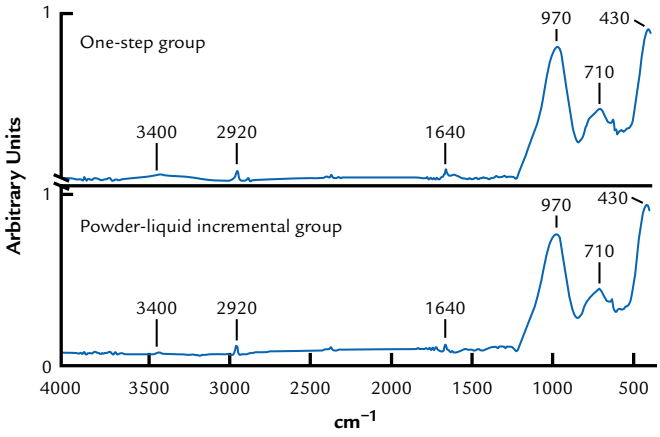
Statistical analysis was conducted using a descriptive approach based in the maximum and minimum values of calculated biaxial flexural strength. The Kolmogorov-Smirnov (KS) test was applied to determine if



1 Characteristic SEM images of specimens polished and HF 2% treated. A, Powder-liquid incremental and B, one-step mixing groups (x2400).



2 X-ray diffraction spectra of sintered dental porcelain discs produced by 2 different mixing methods: a) one-step group and b) powder-liquid incremental group.



3 Fourier transformed infrared attenuated reflectance spectra (absorbance mode) of sintered dental porcelain discs produced by 2 different mixing methods.

the Weibull model was an adequate distribution for the experimental data obtained for both groups. The estimation of the Weibull parameters (m, σ_0) was calculated by the maximum likelihood method using statistical software (R, v2.11.1, R Foundation for Statistical Computing, Vienna, Austria).[40-42] Weibull modulus, characteristic strength, relative humidity and porosity values were compared using 2-tailed Student's t-test or Mann-Whitney test, depending on the evaluated normality of distribution according to KS test. All comparisons were conducted at α =.05.

RESULTS

Descriptive statistical analysis showed that mean (SD) biaxial strength values were 74.61 (12.71) MPa for the powder-liquid incremental mixture group and 69.72 (16.35) MPa for the one-step group. To test that the experimental data fit a Weibull distribution, the data were compared with a theoretical distribution and both groups showed no statistically significant deviations from the theoretical distributions *P*=.528 for one step process and *P*=.685 for incremental process), confirming that the data could be modeled as a Weibull distribution.

The Weibull moduli (SD) obtained were relatively low but the values for the powder-liquid incremental mixing group (6.74 (0.70)) were significantly higher (*P*<.001) than those obtained for the one-step mixing group (4.94 (0.94)). In addition, the characteristic strength (SD) values found were also significantly higher (*P*<.001) for the powder-liquid incremental mixing group (79.87 (2.01) MPa) than for the one-step mixing group (75.95 (2.61) MPa).

The results of chemical analysis by x-ray fluorescence confirmed that the raw porcelain powder has the typical composition of conventional den-

tal porcelains, that is 60% SiO_2; 15% Al_2O_3; 8% K_2O and 6% Na_2O, with smaller amounts of other oxides such as CaO, TiO_2, MgO, SnO_2, BaO and Li_2O that were confirmed by atomic absorption spectroscopy. Significantly higher (P=.009) mean (SD) values of the relative humidity in green-state discs after demold were found for the one-step group (22% (1%)) compared to the powder-liquid incremental mixing group (20% (0.5%)).

No obvious morphological differences were observed in the SEM analysis between fractured discs derived from both preparation methods. In the evaluation of the polished sections (Fig. 1), a small qualitative difference was noticed in the porosity. However, no significant differences (P=.916) were found in the porosity volume test (full data not shown) between both experimental groups.

Figure 2 shows that XRD characteristic spectrum for both groups exhibited the same diffraction pattern of the crystalline phase (identified as cubic Leucite). In addition, a larger amount of amorphous phase was detected in discs produced by one-step mixing. Figure 3 shows a comparison of the FTIR attenuated reflectance spectra for powder-liquid incremental and one-step mixing groups. IR absorption bands with peaks at 430, 710, 970, 1640, 2920 cm^{-1}; as well as a broad absorption band from 3150 to 3650 cm^{-1}, were observed for both groups.

DISCUSSION

The research hypothesis that significant differences would be found between the Weibull parameters calculated from biaxial flexural strength of discs produced by 2 different slurry preparation methods was accepted, since the powder-liquid incremental method resulted in significantly higher Weibull modulus and characteristic strength compared to the one-step technique. The higher Weibull modulus obtained by the powder-liquid incremental method indicates a lower variability of strength values in this group and consequently higher reliability of the structures produced by this technique. The higher characteristic strength obtained for the incremental group indicates that higher stress levels are necessary to fracture 63.2% of the specimens produced using this technique.

The better mechanical behavior of the porcelain specimens produced by the powder-liquid incremental technique is most likely due to the fact that this method results in a lower amount of pores in the porcelain structure compared to the one-step method. The relative humidity analysis showed that higher amounts of liquid were found in the green-state discs of the one-step group. Therefore, it is clear that when water is added in a single step to the porcelain powder, larger amounts of liquid are retained in the spaces between the powder particles. As a consequence, higher porosity is expected due to the larger amount of space between powder particles following the evaporation of the water, reducing the efficacy of the viscous flow sintering process and resulting in higher porosity levels.[23,25]

The porosity level may not be the sole factor affecting the flexural strength of dental ceramics, since a previous study[39] showed that glass-ceramics with significantly higher porosity (due to processing at higher temperatures) possessed statistically similar flexural strengths to glass-ceramics with lower porosity levels. It is important to consider that the flexural strength of brittle materials is determined by the flaw population of the tested material, and therefore, the present study showed that the one-step technique produced in a less favorable flaw population resulted in decreased mechanical behavior.

Another possible explanation for better mechanical behavior of specimens produced by the incremental group is the lower amount of amorphous phase in these specimens, as shown in the XRD analysis. The amorphous phase has been shown to be the weakest part of dental porcelains, since they are more susceptible to water-assisted degradation.[36] Also, a low amount of amorphous phase indicates that other phases are more abundant, such as the leucite phase, which can increase the fracture toughness of dental porcelains by the crack deflection toughening mechanism.[11] It is not obvious how the water content of the green-state specimen affects the phase distribution in the final material. The sintering of dental porcelain within the furnace is a complex viscous flow process and the crystallization of leucite within the glassy matrix is governed by many factors such as temperature, heating rate, and powder characteristics.[38] After sintering, FT-IR spectroscopy was used to evaluate the presence of hydroxyl and molecular water in the bulk discs, since their presence has been associated with a increased relaxation of the glass structure.[26] A broad band with a peak at 3400 cm^{-1} appeared higher in the one-step group compared to the incremental group, which might be interpreted as a higher level of hydroxyl water. However, further studies are necessary to confirm this trend and to understand how excess water may affect the crystalline content of these materials. Other IR absorption peaks and bands showed comparable results for discs from both mixture techniques evaluated. They were determined to correspond to H-O-H bonds (1640 cm^{-1}),[12,36] Si-O-Si bonds (710 cm^{-1}, the bridging vibration of oxygen)[12,36] within the glass network, O-Al-O bonds (430 cm^{-1} corresponds to vibration of oxygen),[37] Si-O- associated with alkaline ions, such as Na^+, K^+ and Ca^{2+} in the glass network (a broad peak from 760 to 970 cm^{-1} results from the combined vibrations of non-bridging oxygen),[37] and O-C-O bonds (2920 cm^{-1} corresponds to vibration of oxygen), a group normally observed in this technique.

Authors of another study[19] that used a different methodology to vary the amount of water added to the porcelain powder presented results

that are in agreement with this study, since it was demonstrated that feldspathic porcelain (Vitadur-Alpha) had its Weibull modulus reduced when a higher amount of water than an optimal powder-liquid ratio of 2.8 was added to the porcelain powders. The same authors[25] studied aluminous porcelain (Vitadur-N) and found that deviating from an optimal powder-liquid ratio (either higher or lower) resulted in a decreased Weibull modulus. These authors indicated that it is important to produce porcelains with relatively low apparent porosity to reduce the amount of shrinkage on firing, and as a consequence, improve the marginal fit of porcelain in practice.

In the present study, the powder/liquid ratio for specimen preparation was selected according to other reports so that one-step mixing group results could be compared with those from other studies.[15,19] Absorbent paper towels were used to remove excess water in the ceramic paste for both groups.[34] Removal of the liquid with an absorbent tissue allows the particles to move closer together under the action of the surface tension of the liquid.[19] Such powder condensation aims at achieving the maximum packing density for the powder, and light vibration techniques are used to reorient the powder particles into a position of maximum packing efficiency by eliminating as much excess liquid as possible.[19,25] However, this procedure also added a further variability in the preparation of green-state discs. Another aspect related with specimen preparation in the present study is that the discs were polished and not glazed to better control the final disc thickness and to reduce the complexity of the studied model; however, this procedure probably exposed pores and increased surface roughness.[32]

The results of the present study are clinically relevant because they showed that dental technicians should be encouraged to use the powder-liquid incremental technique presented for the production of ceramic prostheses with higher structural homogeneity and better mechanical properties. However, some limitations of the present work should be considered, such as the use of only one commercial porcelain powder, as different results may be obtained with different porcelains or ceramic compositions. Also, the number of specimens could have been increased to improve the power of the characterization by high resolution techniques. Finally, direct extrapolation to the clinical situation should be carefully considered as this study did not investigate other factors present in the oral cavity such as pH variations, thermal cycling or occlusal forces.

CONCLUSIONS

Considering the limitations of this in vitro study, the results showed that the incremental technique for the preparation of porcelain slurry resulted in lower water content in the green-state specimens, and compared to the one-step technique, produced significant improvements in both the Weibull modulus and characteristic strength of the porcelain tested. Microstructural analyses showed that observed improvements in the mechanical properties of the specimens prepared by the incremental technique may be related to decreased porosity and increased crystalline content.

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NOTEWORTHY ABSTRACTS OF THE CURRENT LITERATURE

A prospective 15-year evaluation of extensive dentin-enamel-bonded pressed ceramic coverages

van Dijken JW, Hasselrot L.
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Objectives. The purpose of this study was to investigate the durability of extensive dentin-enamel-bonded posterior ceramic coverages in a 15 years follow-up.

Methods. All extensive dentin-enamel-bonded posterior partial and complete all-ceramic coverages placed during the period November 1992–December 1998 were included. In 121 patients, 252 coverages (IPS Empress) were placed. The adhesive bonding to dentin and enamel was performed with three 3-step and one 2-step etch and rinse bonding. In 106 restorations the classic Syntac was used in combination with the dual-cured resin composite Variolink. The other restorations were luted with the chemically cured resin composite Bisfil 2B and bonded with 3-step etch and rinse systems, classic Gluma (37), Allbond 2 (57), Syntac (32) or the 2-step etch and rinse system, One step (20). The ceramics were evaluated yearly by modified USPHS criteria during 15 years.

Results. Postoperative sensitivity was registered in 4 patients during bite forces lasting for 2–4 weeks. Fifty-five of 228 coverages (24.1%) failed. The mean observation period of the acceptable coverages was 12.6 years (range 11–15 years). The main reasons for failure were lost restorations (18), ceramic fracture (16), and secondary caries (11). Significant differences in failure rate were observed between the dentin bonding agents but not between the two luting agents. Ceramic coverages placed on non-vital teeth failed in 39% and on vital teeth in 20.9% ($p = 0.014$). Logistic regression indicated three significant predictors for failure of the coverages: gender and parafunctional habits of the patient and non-vitality of the tooth.

Significance. The technique investigated showed advantages like less destruction of healthy tissue, and avoiding of endodontic treatment and/or deep cervical placement of restoration margins to obtain retention.