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Effects of acid treatment with ultrasonic on resin-zirconia bond strength

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Introduction

Nowadays zirconium oxide has been widely used in restorative dentistry due to its biocompatibility, esthetics, and mechanical properties. Zirconium oxide coping for fixed partial denture framework requires the application of veneering porcelain and resin based cement as well. To promote micromechanical interlocking, many surface roughening methods for zirconia were developed. Hydrofluoric acid (HF) treatment is commonly used on silica-based ceramics to generate surface roughness. Recently, due to its resistance to conventional etching techniques, alternative surface etching technique of zirconia coping has been introduced to increase the surface roughness. And it would also be useful to increase the bond strength of resin cement. Casucci et al. found that hot acid etching increased the bond strength between zirconia and resins. Mechanical properties of zirconia were not affected, which in turn was due to its low handling temperature. [1, 2] In addition, zirconium oxide etched with hot H_2SO_4/HF/HNO_3 and HF/HNO_3 created excellent three-dimensional networks. [3] Ultrasonic would play an important role in molecular motion that more easily the protons become ionized, more acidic the outcomes. Therefore, zirconia could be dissolved effectively in acids with ultrasonic.

Purpose of the Study

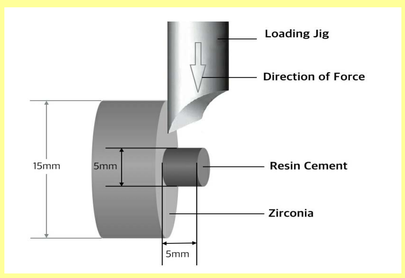
The purpose of this study is to investigate shear bond strength of resin cements on etched / non-etched zirconium oxide block. The null hypothesis of this study was that the acid etching with ultrasonic does not affect shear bond strength values between zirconium oxide and resin cements.

Materials and Methods

Zirconia core material (ACUCERA Inc., Korea) and two resin cements were selected. Rely X Unicem U200 (3M Deutschland GmbH, Germany) and Multilink N (Ivoclar Vivadent AG, Liechtenstein) were used. Forty-eight Y-TZP (yttria-tetragonal zirconia polycrystals) discs (Diameter 15mm, Height 4mm) were produced and mirror-like finished and polished. Y-TZP discs were randomly assigned to four groups (n=12 per group) according to the surface treatment and resin cement used. Four experimental groups were shown as follows: Multilink on acid-etched surface (M/E), Unicem on acid-etched surface (U/E), Multilink on polished surface (M/P), Unicem on polished surface (U/P).

Half of the specimens were dipped in HF/HNO_3 solution (ZIRCOS-E, Bioden co., Korea) [Fig. 1] and kept for 30 min with 5 min ultrasonic. After rinsing with distilled water and steam cleaning, annealing processes were subsequently conducted at 1150ºC. For the shear bond strength test resin cements (Diameter 6mm, Height 5mm) were bonded on the Y-TZP surface

[Fig 1] ZIRCOS-E, Bioden.co., Korea



[Fig 2] Illustration of the SBS testing.

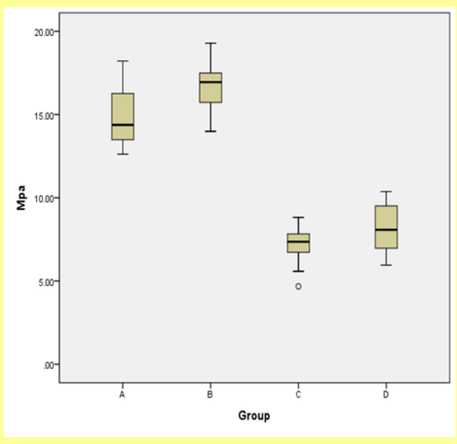
using putty index according to manufacturers' recommendation.

Surface roughness of 3 specimens of each group was evaluated using a 3D optical analyzer (Burker, Daltonik, Germany), and microscope analysis was performed. Bonded specimens were subjected to shear bond strength (SBS) test at a crosshead speed of 1mm/min using a universal testing machine (Instron Model 3366, USA). [Fig. 2]

After shear bond strength test, all fractured surfaces of specimens were examined to determine the failure pattern by a digital microscope using X10 magnification. Fracture modes were classified as adhesive failure (fracture occurring at resin cement and zirconia interface), cohesive failure (fracture occurring within resin cement), or mixed failure (occurrence of both adhesive failure and cohesive failure). Statistical analyses were performed using the statistical software IBM SPSS Statistics 22. The Levene's test was applied to ensure the normal distribution of data before the ANOVA ($p<.05$). The one-way ANOVA was performed for each group to determine the difference of shear bond strength values ($p=0.05$).

Results and Discussion

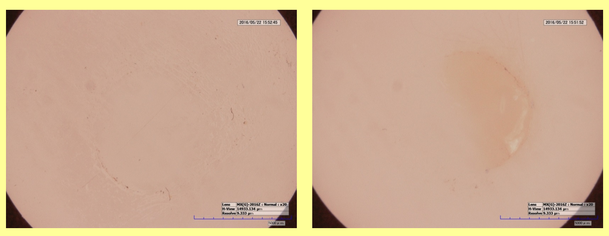
The mean shear bond strength values and standard deviations of each group are provided in Table 1 and shown in a box plot. [Fig. 3] The mean±standard deviation of shear bond strength were 14.9 ± 1.9 MPa in U/E, 16.6 ± 1.4 MPa in M/E, 7.2 ± 1.1 MPa in U/P, 8.2 ± 1.5 MPa in M/P group, respectively, and were significant different ($p<0.05$). The average shear bond strength was largest in M/E group, followed by U/E, M/P, and U/P groups.



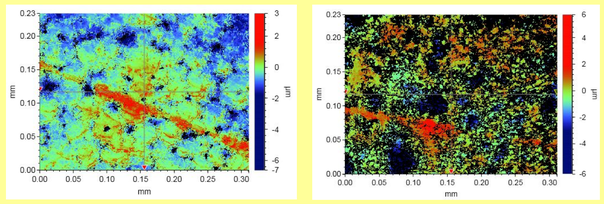
[Fig 3] Box-plot diagram of the shear bond strength (Mpa)

	Group U/E(A)	Group M/E(B)	Group U/P(C)	Group M/P(D)
Mean(Mpa)	14.9	16.6	7.2	8.2
SD	1.9	1.4	1.1	1.5

[Table 1] The shear bond strength values of each group



[Fig 4] Adhesive failure (left) vs mixed failure (right).



[Fig 5] 3D optical images of polished (left) and HF/HNO_3 etched with ultrasonic (right).

Most specimens in polishing groups (U/P and M/P) showed adhesive failure mode and most specimens in etching groups (U/E and M/E) showed mixed failure mode at the interface between zirconia and resin cements. [Fig. 4] 3D optical images of polished and etched surface of zirconia are shown in Fig. 5. Acid etching with ultrasonic resulted in rough textures, while a relatively flat morphology at a micron scale compared with mirror-like polishing. Etching with HF/HNO_3 opened the intergrain spaces and revealing a porous three-dimensional network. [3]

Conclusion

Zirconia is hardly dissolved by hydrofluoric acid (HF) treatment, commonly used on silica-based ceramics. Alternative surface etching technique of zirconia coping is introduced to increase the surface roughness. A combination of acid etching with HF/HNO_3 and ultrasonic seemed to be more promising to improve bonding performance of resin-based cements. Within the limitations of this study, compared to non-etching, acid etching with ultrasonic improved the shear bond strength between zirconia and resin cements.

References

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