Surface wettability and nano roughness at different grit blasting operational pressures and their effects on resin cement to zirconia adhesion

Aftab Ahmed KHAN1, Badreddin Abdelhaman MOHAMED2, Eraj Humayun MIRZA3, Jamaluddin SYED4, Darshan Devang DIVAKAR1 and Pekka Kalevi VALLITTU5,6,7

1 Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia
2 Community Health Department, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia
3 Department of Biomedical Engineering, NED University of Engineering & Technology, Karachi, Pakistan
4 Oral Basic and Clinical Sciences Department, Faculty of Dentistry, King Abdul Aziz University, Jeddah, Saudi Arabia
5 Department of Biomaterials Science, Institute of Dentistry, University of Turku, Finland
6 City of Turku, Welfare Division, Turku, Finland
7 Visiting professor King Saud University, Riyadh, Saudi Arabia

Corresponding author, Aftab Ahmed KHAN; E-mail: aakjk@hotmail.com

INTRODUCTION

The toxic and allergic reactions to some alloys led to the decline in the popularity of metal or metal-ceramic restorations31. All-ceramic restorations were proposed as an alternative to metal or metal-ceramic restorations because of their better chemical stability, biocompatibility, natural-looking shade, and appearance32. However, brittleness and low tensile strength limited their application as resilience and high tensile strength are deemed necessary for any restorative material33. Weakness of a material can be overcome to certain extent, especially in single-unit restorations, by good adhesion to the underlying dental tissues. However, in multiple-unit fixed dental prostheses, the durability of a restorative material is a key factor for the clinical success of the restoration6. The recent advent of advanced dental ceramics, i.e., zirconium di-oxide or zirconia has introduced a new chapter in dentistry in terms of superior fracture strength and toughness.

Zirconia (ZrO2) is a polymorphic material that can be transformed from one crystalline phase to another during sintering34. Zirconia’s usefulness in dentistry comes from its “stabilized” phase. Investigators have found that when ZrO2 is blended with varying amounts of lower valence oxides such as calcium oxide (CaO), magnesium oxide (MgO), and yttrium oxide (Y2O3), it allows the formation of partially or fully stabilized ZrO2. The resultant ceramic may have exceptional physical, chemical, and mechanical properties6.

Today, yttria (Y2O3)-stabilized polycrystalline tetragonal zirconia (Y-TZP) is a frequently used ceramic material for fixed prosthodontics, largely because of its favorable mechanical properties35,36. However, the relative inertness of ZrO2 renders its surface less reactive7,9). Various surface treatment methods have been advocated to roughen and/or activate the ZrO2 surface for adhesion with resin cements8,10). Grit blasting with silica-coated grit particles results in the embedding of the silica content on the ceramic surface, rendering the silica-modified surface chemically reactive to the resin through silane coupling agents2,12). In contrast, the operating air pressure of silica-coating as an alternative treatment method to promote adhesion with resin cement10,11). Grit blasting with silica-coated grit particles results in the embedding of the silica content on the ceramic surface, rendering the silica-modified surface chemically reactive to the resin through silane coupling agents2,12). In contrast, the operating air pressure of silica-coating as an alternative treatment method to promote adhesion with resin cement10,11).

Data suggesting that by increasing the operating air pressure of tribochemical silica coating system, the adhesion strength of resin cement to zirconia might be improved. However, the mechanism behind the improvement is still not known. It may be related to changes in the surface roughness and wettability by increasing the operational air pressure. Therefore, the aims of this study were to investigate the effects...
of operational air pressure of the tribochemical silica coating system on the surface roughness and surface wettability. Secondly, this study also aimed to assess the possible correlation of these independent variables to the adhesion of resin cement to zirconia. The null hypothesis of this study was that there would be no change in the adhesion strength of resin cement to zirconia.

**MATERIALS AND METHODS**

**Specimen preparation**

The pre-sintered, yttrium-stabilized zirconium di-oxide (ZS blanks; KaVo Everest®, KaVo Dental, Biberach, Germany) with ≤94 ZrO₂ purity were obtained from the manufacturer and longitudinally cut into three equal halves with a saw blade. Sintering was performed according to the manufacturer’s instructions in a calibrated furnace (KaVo Everest engine, KaVo Dental). Then the ZrO₂ blanks were embedded into plastic cylinders with an acrylic resin base material (Palapress™, Heraeus Kulzer, Hanau, Germany), leaving one surface free for adhesion. The surfaces of all blanks were finished by wet-grinding with 600-grit silicon carbide abrasive paper and cleaned in 70.0% ethanol solution for 5 min in an ultrasonicator. The upper halves of these ZrO₂ blanks were coated with tribochemical silica-coated powder (Rocatec™ Soft, 3M ESPE, Seefeld, Germany). The distance, angulation and grit-blasting time were followed according to the manufacturer’s instructions, i.e., with a slowly rotating motion in a jet from a perpendicular distance of 10 mm to the zirconia surface for 10 s. However, the grit blasting air pressures were as follows: group 1: no treatment (control), group 2: specimens were treated at 80 kPa, group 3: specimens were treated at 180 kPa, group 4: specimens were treated at 280 kPa, and group 5: specimens were treated at 380 kPa. The ZrO₂ blanks were again cleansed ultrasonically in 70.0% ethanol solution for 5 min and air dried.

A single coat of commercially available silane primer (Monobond® N, Ivoclar Vivadent, Schaan, Liechtenstein) was applied with a primer applicator brush on the silica-coated ZrO₂ surface in all the groups and left for 3 min to air dry. Before the adhesion process, a semi-transparent polystyrene-enclosed mold was fixed on the surface of the specimen (2.40 mm, 4.5 mm high). Self-adhesive resin cement (Multilink® Speed, Ivoclar Vivadent) was prepared according to the manufacturer’s instructions and filled into the enclosed mold. Eight resin stubs were adhered to each ZrO₂ blank. Each resin stub was polymerized for 30 s from the top of the mold and then from the lateral side for 30 s using a light-curing unit (Elipar™ 2500, 3M ESPE, St Paul, MN, USA) with the light intensity of 600 mW/cm². The resin stubs adhered to the ZrO₂ substrate were engaged perpendicular to the round-notched blade and loaded onto a universal testing machine (Model no. 3369 Instron, Canton, MA, USA) with a cross head speed of 1 mm/min until failure. The EM-SBS was calculated using the following equation:

\[ S = \frac{1}{L/A} \]  

Where, L=applied load at failure in Newton (N), and A=adhesive area of the specimen in mm².

**Scanning electron microscopy (SEM) analysis**

The impact of surface treatment after silica-coating at different grit blasting air pressures was evaluated by SEM (JSM 5500, JEOL, Tokyo, Japan). The specimens were first mounted onto an aluminum specimen-holder. Subsequently, SEM analysis was performed at an operating voltage of 10 kV in the secondary electrons (SE) mode and the images were captured using the proprietary software of the SEM system. Magnifications of 100, 1,000, and 10,000 were used to observe the surface morphology.

**Atomic force microscopy (AFM) analysis**

A single representative specimen from each operational air pressure group was subjected to surface topographical analysis. AFM (Multimode8-U, Bruker, Santa Barbara, CA, USA) was used to analyze the average roughness (Rₐ) of the specimens. Scanning of the treated ZrO₂ surfaces was performed in tapping mode using a beam-type cantilever, antimony n-doped Si tip (RTESP-300, nominal spring constant 40 N/m). The oscillating frequency was set at 170 kHz. A set of five frames of the same sizes (4.8×4.8 μm) was taken from different areas of the surface of the specimen with a slow scan rate of 1 Hz.

**Enclosed mold shear bond strength (EM-SBS) testing**

The resin stubs adhered to the ZrO₂ substrate were engaged perpendicular to the round-notched blade and loaded onto a universal testing machine (Model no. 3369 Instron, Canton, MA, USA) with a cross head speed of 1 mm/min until failure. The EM-SBS was calculated using the following equation:

\[ S = \frac{1}{L/A} \]  

Where, L=applied load at failure in Newton (N), and A=adhesive area of the specimen in mm².

**Fractographic investigation**

Fractured or debonded specimen surfaces were examined using a stereomicroscope (Nikon SM2-10, Tokyo, Japan) at 40x magnification. The fractured surfaces were assigned to the following three failure patterns: cohesive failure within the composite resin restorative material or resin cement; adhesive failure at the ceramic/cement interface; or mixed failure, i.e., both adhesive and cohesive failure modes.
Statistical analysis
One-way analysis of variance (ANOVA) and two-way ANOVA were selected to analyze the data with one within-subject factor (storage conditions, three levels) and one between-subjects factor (ZrO₂ surface treatment, five levels). The Tukey’s post hoc test was selected for pairwise comparisons (p=0.05). Pearson’s correlation between the contact angle and surface roughness was determined with respect to grit blasting pressure. Furthermore, correlation between EM-SBS and contact angle at different grit blasting air pressures was analyzed. All data were analyzed digitally, using SPSS 21.0 software (SPSS, Chicago, IL, USA).

RESULTS

SEM
Figure 1 shows the morphological changes of the ZrO₂ substrate at different grit blasting operational pressures. The abrasive roughness created by 600-grit...
silicon carbide paper in the control group can be seen in Figs. 1A–C. In contrast, tribochemical-treated surfaces showed modified surface textures with increased surface roughness (Figs. 1F, I, L, and O). The gravity of cracks and micro-mechanical grooves were the consequences of increased operational grit blasting pressures.

**Surface roughness**
The quantitatively examined surface topography by AFM is presented in Table 1. The representative specimen of the control group showed a relatively smooth surface, with the lowest surface roughness value (35.2±4.5 nm). However, the ZrO₂ surfaces treated at different grit blasting air pressures revealed relatively uneven and irregular surfaces, which increased with increasing grit blasting pressures. The AFM images of the study groups are presented in Fig. 2.

**Table 1  The surface roughness (Ra) and the corresponding contact angle (θ) of the tested surfaces**

| Group   | Surface roughness (Ra) (nm) | Contact angle (θ) (°) |
|---------|-----------------------------|-----------------------|
| Control | 35.2±4.5<sup>A,B,C</sup>   | 83.2±4.2<sup>A,B,C,D</sup> |
| 80 kPa  | 36.3±3.6<sup>D,E,F</sup>   | 65.7±3.8<sup>A,E,F,G</sup> |
| 180 kPa | 60.9±4.3<sup>A,D,G</sup>   | 57.3±4.1<sup>B,E,H</sup> |
| 280 kPa | 81.0±7.1<sup>B,E,H</sup>   | 53.6±3.6<sup>C,F</sup>   |
| 380 kPa | 109.3±9.3<sup>C,F,G,H</sup> | 49.2±4.4<sup>D,G,H</sup> |

Ra=arithmetic average of surface roughness. Same uppercase letters show statistical difference between the grit blasting pressure groups.

**Fig. 2** AFM images of surface modified zirconia. (A) 600-grit silicon carbide abrasive paper; (B) Rocatec™ Soft used at 80 kPa; (C) Rocatec™ Soft used at 180 kPa; (D) Rocatec™ Soft used at 280 kPa; (E) Rocatec™ Soft used at 380 kPa.
Contact angle
ZrO2, whose surface was modified with a 600-grit silicon carbide abrasive paper, showed the highest water contact angle of 83.2°±4.2°. However, grit blasting affected the surface properties of ZrO2 and made the surface hydrophilic (Table 1). The lowest water contact angle was observed in 380 kPa group (49.2°±4.4°).

Adhesion (EM-SBS) strength
The mean EM-SBS values for the five surface procedures and three storage/aging conditions are presented in Table 2. The results revealed significant increase in the adhesion (bond) strength values with the increasing grit blasting pressure following 24 h of dry storage, i.e., the control group had a mean SBS of 7.6±1.7 MPa whereas the 380 kPa group had a mean SBS of 15.7±5.3 MPa. Excluding the control group, the experimental groups subjected to artificial water aging for 6,000 thermo-cycles achieved greater SBS than the 24 h dry storage groups. However, 12,000 thermo-cycles had a diminutive impact on the SBS values. The control group achieved the lowest SBS value (5.9±4.0 MPa) whereas the 280 kPa group had the highest EM-SBS (19.4±4.8 MPa). The graphical representation of EM-SBS is shown in Fig. 3.

The result of one-way ANOVA showed statistically insignificant difference between the aging groups. However, two-way ANOVA and the Tukey’s post hoc test revealed a significant correlation between the grit blasting pressure groups. Pearson’s correlation results further revealed that the increase in surface nano-roughness due to higher grit blasting air pressure has a negative influence on the surface wettability of a zirconia substrate (r=−0.906, p=0.01).

Table 2 Enclosed mold shear bond strength means (in MPa) and standard deviations of the study groups with percentage of enhancement

| Storage condition   | Group | Mean±SD (MPa) | Change in SBS (%) |
|---------------------|-------|---------------|-------------------|
| 24 h dry storage    | Control | 7.6±1.7       | 0                 |
|                     | 80 kPa  | 11.7±1.9      | 53.9              |
|                     | 180 kPa | 14.6±4.4      | 92.1              |
|                     | 280 kPa | 14.0±1.8      | 84.2              |
|                     | 380 kPa | 15.7±5.3      | 106.5             |
|                     | Control | 6.3±2.0       | 0                 |
|                     | 80 kPa  | 14.7±3.5      | 133.3             |
| 6,000 thermo-cycles | 180 kPa | 21.0±3.6      | 233.3             |
|                     | 280 kPa | 21.4±4.3      | 239.6             |
|                     | 380 kPa | 16.2±2.9      | 157.1             |
|                     | Control | 5.9±4.0       | 0                 |
|                     | 80 kPa  | 9.3±3.3       | 57.6              |
| 12,000 thermo-cycles| 180 kPa | 17.4±6.7      | 194.9             |
|                     | 280 kPa | 19.4±4.8      | 228.8             |
|                     | 380 kPa | 14.8±4.8      | 150.8             |

Fig. 3 Bar chart representation of the EM-SBS values of the study groups with error bars indicating standard deviations (n=8). Same uppercase letters show statistical difference between the grit blasting pressure groups.
Fractography results
The visual examination of the failure mode showed mainly mixed and adhesive fractures (Table 3). Few pure resin composite cohesive failures were observed only in the 380 kPa group after 24 h dry storage. However, after 12,000 thermal cycles, the 380 kPa group did not show any cohesive fracture. In addition, cohesive fractures were observed in the 180 and 280 kPa groups, after 12,000 thermo-cycles. Excluding the specimens of the control and the 80 kPa groups, which showed only adhesive fractures, the remaining study groups exhibited a combination of two or more than two failure modes under all the aging conditions.

DISCUSSION
In this study, KaVo ZrO₂ blanks were chosen because it is one of the established suppliers of Y-TZP ZrO₂-based ceramics. Tribochemical silicoating with Rocatec™ Soft at higher grit blasting operational pressures significantly improved the adhesion of resin cement to ZrO₂. The EM-SBS results of this study are in accordance with the work of Heikkinen et al., who believed that the adhesion strength of resin cement to ZrO₂ is correlated to the air pressure of grit blasting⁵⁰. However, a slight variation in the results could be attributed to factors such as the operator’s hand, aging conditions, and experimental protocols. Since adhesion (enclosed mold shear bond) strength improved with the pre-treatment, our null hypothesis is rejected.

The bigger grit particles were anticipated to produce microcracks on ZrO₂ substrates, which may become the sources of stress raisers. Therefore, Rocatec™ Soft (30 µm silica-coated alumina particle) powder was selected. Previous studies have advocated tribochemical silica coating as an effective pre-treatment method for promoting the adhesion of resin cement to ZrO₂. We observed that the surface roughness generated by the grit blasting operational pressures had a direct correlation with the adhesion strength of the resin cement to ZrO₂ after 24 h dry storage. However, the pivotal role of the surface roughness was difficult to infer in artificial water-aged specimens, since no correlation was observed. The reason could be the flaws and microcracks that originate at elevated operational pressures. Damaged surface leads to reduction of the elastic modulus and surface loss⁵¹.⁵².

The continuous immersion in water further deteriorates the damaged surface, and causes material loss around the microcracks. Here, it is imperative to mention that the thickness of a ZrO₂ coping is in the range of 0.5 to 0.7 mm. Grit blasting at elevated operational pressures could have a far-reaching effect on the clinical longevity of a ZrO₂ restoration. Flaws and surface defects may adversely affect the surface adhesion and the longevity of a restoration.

The invasive effect on ZrO₂ substrate was confirmed when the representative specimens were qualitatively analyzed under SEM. Different surface irregularities and attached silica particles can be seen in Fig. 1. An increased number of microcracks can be observed at higher operational pressure of grit blasting (Figs. 1K and N). Concurrent to these surface changes, the growing content of silica deposition can also be observed at higher grit blasting pressures (Figs. 1L and O). The effect of 600-grit silicon carbide abrasive paper on ZrO₂ is also evident in Figs. 1A–C.

As expected, the representative specimen for AFM analysis revealed that ZrO₂ surface was moderately roughened even at higher grit blasting pressures. The highest surface roughness value was observed in the 380 kPa group (109 nm). The diminutive, damaging effect of the grit blasting air pressure of 380 kPa could be due to the smaller grit particles, i.e., 30 µm in size. Larger grit particles might have a greater detrimental effect on the ZrO₂ surface. The visual interpretation of the 380 kPa operational pressure effect can be seen in a 3D image (Fig. 2E). Dissimilarity in surface textures was evident according to the different surface treatment methods used (Fig. 2). Both SEM and AFM data were in agreement with each other and showed a relative effect of the grit blasting operational pressures on the surface properties of ZrO₂.

The contact angle measurements further affirmed the effect of grit blasting operational pressures on the surface properties. Distilled water was selected for the measurement of contact angle because it has low viscosity and vapor pressure. All the experimental groups presented a gradual depletion in the contact angle with increasing grit blasting operational pressures, indicating increased wettability and potential surface reactivity with resin based cement. The increased wetting or hydrophilicity in the 380 kPa group was

Table 3 Stereomicroscope classification of the failure modes

| Storage condition | Grit blasting pressure | Control | 80 kPa | 180 kPa | 280 kPa | 380 kPa |
|------------------|------------------------|---------|--------|---------|---------|---------|
|                  |                        | A       | M      | C       | A       | M      | C       | A       | M      | C       | A       | M      | C       |
| 24 h dry storage |                        | 100     | 0      | 0       | 100     | 0      | 0       | 75      | 25     | 0       | 75      | 25     | 0       | 62.5    | 25     | 12.5    |
| 6,000 thermo-cycles |                  | 100     | 0      | 0       | 100     | 0      | 0       | 50      | 25     | 25      | 62.5    | 25     | 12.5    | 62.5    | 25     | 12.5    |
| 12,000 thermo-cycles |                | 100     | 0      | 0       | 100     | 0      | 0       | 62.5    | 25     | 12.5    | 62.5    | 25     | 12.5    | 75      | 25     | 0       |

A=adhesive fracture, M=mixed fracture, C=cohesive fracture
observed due to increased surface roughness (Table 1).

The initial drastic reduction in contact angles from the control to the 180 kPa groups and then marginal differences in the contact angles of the 180, 280, and 380 kPa groups could be attributed to silicatization of the surface by Rocatec™ Soft grit blasting system: silica-coverage reached the maximum at 180 kPa pressure and silica-coating due to its spontaneous OH-coverage on ZrO2 substrate became hydrophilic, increasing its wettability. In addition, a correlation between EM-SBS and the contact angles was observed, which showed that wetting behavior and EM-SBS are not correlated ($r=−0.542, p=0.01$).

A semi-transparent polystyrene-enclosed mold was used to prepare the resin cement stubs. Keeping in view the initial adhesion strength of resin cement to ZrO2, the molds were not disengaged after polymerization of the resin stubs[23,24]. Monobond N was used to prime the ZrO2 surface. Monobond N contains methacrylated phosphoric acid ester (1–<2.5%). The phosphate monomer is a time-tested and commonly used monomer which has a special affinity for silica-free ceramics[25,26]. All the experimental groups showed increased adhesion (enclosed mold shear) strength after 6,000 thermal cycles, except the control group. This could be attributed to further polymerization of the monomer by the repeated exposure at an elevated temperature of 55°C in thermal-cycling or due to release of stresses generated during the polymerization contraction of composite cement. In addition, it may also be possible that lesser expansion of the polymeric material at 55°C had resulted in a water-tight interface that led to higher EM-SBS values in the 6,000 thermal cycling groups. However, at the end of 12,000 cycles, decreased EM-SBS values were observed in all the groups (Fig. 3). The detrimental effect of water might have contributed to the fatigue of the siloxane bond due to the differences in thermal expansion coefficient[27,28].

Artificial water aging cannot truly mimic the clinical conditions where other physiological factors (masticatory forces, salivary pH etc.) are involved. Their results, therefore, cannot be correlated with the duration of clinical service. Nevertheless, artificial water aging regimens are considered to be vital for any laboratory-based study[29]. Although no benchmark is set for the adequate adhesion strength for resin composite cement to all-ceramic materials, investigators have suggested that adhesion strength ranging from 10 to 13 MPa are acceptable for clinical service[22,23]. After 12,000 thermal-cycles, the 180, 280, and 380 kPa groups achieved adhesion strengths greater than the suggested range, which means that favorable SBS results may be achieved either by using 180, 280, or 380 kPa grit-blasting air pressures. The depleting EM-SBS values in the control group implied that surface treatment is essential for adequate adhesion of the resin cement to ZrO2.

It is necessary to mention here that shear bond test was desired over tensile test because it is easy to perform and quickly indicates the adhesion strength[31]. We investigated the failure modes with a light stereomicroscope. It was not surprising to see the adhesive fractures in the control and 80 kPa groups. Non-silicatized or inadequately silicatized surfaces of the control and 80 kPa groups might be the causes of this type of fracture. Although interfacial fractures were predominant in the rest of the study groups, few cohesive and mixed fractures were noticed. For the 180 kPa group, 25% of the specimens showed cohesive fractures after 6,000 cycles. Whereas, the 280 kPa group showed 12.5% of the cohesive fractures. However, after 12,000 cycles, the bond stability of the 180 kPa group was slightly affected by artificial water aging and we noticed only 12.5% of the cohesive fractures. Whereas, the thermal-cycling induced no effect on the bonding effectiveness of the 280 kPa group.

In this study, thermal-cycler was used for artificial water aging of the specimens. To remove ambiguity, different artificial aging parameters need to be directed for future studies. More consequential results might be attained with longer durations of artificial water aging. In vivo or long-term clinical trials are indispensable to rule out the uncertainty prevailing over ideal grit blasting air pressure.

**CONCLUSIONS**

We infer that:
- Grit blasting at different operational air pressures has both qualitative and quantitative effects on the surface morphology and topography of the ZrO2 substrate.
- Surface roughness of the ZrO2 increased linearly when the operational air pressure was increased whereas surface wettability had a non-linear relationship with the operational air pressure.
- Formation of a low contact angle with a ZrO2 surface is not indispensable to enhancement of adhesion of resin cement to ZrO2.
- Thermal cycling increased the EM-SBS in every experimental group after 6,000 cycles.
- The 180 kPa grit blasting operational air pressure may be used effectively in lieu of recommended the 280 kPa operational air pressure.

**ACKNOWLEDGMENTS**

The authors are grateful to the Deanship of Scientific Research, King Saud University for funding through the Vice Deanship of Scientific Research Chairs.

**CONFLICT OF INTEREST**

The authors declare no conflict of interest.

**REFERENCES**

1. Madfa AA, Al-Sanabani FA, Al-Qudami NH, Al-Sanabani JS, Amran AG. Use of zirconia in dentistry: An overview. Open Biomater 2014; 5: 1-9.
2. Shin YJ, Shin Y, Yi YA, Kim J, Lee IB, Cho BH, Son HH, Seo DG. Evaluation of the shear bond strength of resin cement to Y-TZP ceramic after different surface treatments. Scanning...
3) Shenoy A, Shenoy N. Dental ceramics: An update. J Conserv Dent 2010; 13: 195-204.
4) Marquardt P, Strub JR. Survival rates of IPS empress 2 all-ceramic crowns and fixed partial dentures: Results of a 5-year prospective clinical study. Quintessence Int 2006; 37: 253-259.
5) Camposilvan E, Marro FG, Mestra A, Anglada M. Enhanced reliability of yttria-stabilized zirconia for dental applications. Acta Biomater 2015; 17: 36-46.
6) Piconi C, Maccaruo G. Zirconia as a ceramic biomaterial. Biomaterials 1999; 20: 1-25.
7) Lee MH, Son JS, Kim KH, Kwon TY. Improved resin-zirconia bonding by room temperature hydrofluoric acid etching. Materials 2015; 8: 850-866.
8) Arai M, Takagaki T, Takahashi A, Tagami J. The role of functional phosphoric acid ester monomers in the surface treatment of yttria-stabilized tetragonal zirconia polycrystals. Dent Mater J 2017; 36: 190-194.
9) Ozcan M, Nijhuis H, Valandro LF. Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with MPF functional monomer to zirconia after thermal aging. Dent Mater J 2008; 27: 99-104.
10) Qeblawi DM, Muñoz CA, Brewer JD, Monaco EA. The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. J Prostheth Dent 2010; 103: 210-220.
11) Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. Quintessence Int 2007; 38: 745-753.
12) Cheung G, Botelho MG, Matinlinna JP. Effect of surface treatments of zirconia ceramics on the bond strength to resin cement. J Adhes Dent 2014; 16: 49-56.
13) Heikkinen TT, Lassila LV, Matinlinna JP, Vallittu PK. Effect of operating air pressure on tribochemical silica-coating. Acta Odontol Scand 2007; 65: 241-248.
14) Khan AA, AL Kheraif AA, AL hijji SM, Matinlinna JP. Effect of grit-blasting air pressure on adhesion strength of resin to titanium. Int J Adhes Adhes 2016; 65: 41-46.
15) Khan AA, AL Kheraif AA, Syed J, Divakar DD, Matinlinna JP. Enhanced resin zirconia adhesion with carbon nanotubes-infused silanes: A pilot study. J Adhes 2018; 94: 167-180.
16) Sawada T, Spintzyk S, Schille C, Zöldföldi J, Paterakis A, Schweizer E, Stephan I, Rupp F, Geis-Gerstorfer J. Influence of pre-sintered zirconia surface conditioning on shear bond strength to resin cement. Materials (Basel) 2016; 9: 518-523.
17) Khan AA, AL Kheraif AA, Syed J, Divakar DD, Matinlinna JP. Enhanced resin titanium adhesion with silane primers using tribochemical silica-coating. Dent Mater J 2017; 36: 111-116.
18) Aboushelib MN. Evaluation of zirconia/resin bond strength and interface quality using a new technique. J Adhes Dent 2011; 13: 255-260.
19) Khan AA, AL Kheraif AA, Syed J, Divakar DD, Matinlinna JP. Effect of experimental primers on hydrolytic stability of resin zirconia bonding. J Adhes Sci Technol 2017; 31: 1094-1104.
20) de Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic. Dent Mater 2009; 25: 172-179.
21) Kosmać T, Obling C, Marion L. The effects of dental grinding and sandblasting on ageing and fatigue behavior of dental zirconia (Y-TZP) ceramics. J Eur Ceram Soc 2008; 28: 1085-1090.
22) Su N, Yue L, Liao Y, Liu W, Zhang H, Li X, Wang H, Shen J. The effect of various sandblasting conditions on surface changes of dental zirconia and shear bond strength between zirconia core and indirect composite resin. J Adv Prosthodont 2015; 7: 214-223.
23) Cheetham JJ, Palamara JE, Tyas MJ, Burrow MF. A comparison of the micro-shear bond strength and failure mode of non-enclosed and mold-enclosed luting cements bonded to metal. Dent Mater J 2013; 32: 896-905.
24) Cheetham JJ, Palamara J, Tyas MJ, Burrow MF. A comparison of the shear bond strength and failure mode to metals of unsupported and supported luting cement specimens. J Adhes Dent 2014; 16: 251-260.
25) Kern M. Resin bonding to oxide ceramics for dental restorations. J Adhes Sci Technol 2009; 23: 1097-1111.
26) Thompson JY, Stoner BR, Fiasčk JR, Smith R. Adhesion/ cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater 2011; 27: 71-82.
27) Matinlinna JP, Lassila LV, Vallittu PK. The effect of five silane coupling agents on the bond strength of a luting cement to a silica-coated titanium. Dent Mater 2007; 23: 1173-1180.
28) Heikkinen T, Matinlinna J, Vallittu P, Lassila L. Long term water storage deteriorates bonding of composite resin to alumina and zirconia short communication. Open Dent J 2013; 7: 123-125.
29) Korkmaz Y, Gurgan S, Firat E, Nathanson D. Effect of adhesives and thermocycling on the shear bond strength of a nano-composite to coronal and root dentin. Oper Dent 2010; 35: 522-529.
30) Matsumura H, Yanagida H, Tanoue N, Atsuta M, Shimoe S. Shear bond strength of resin composite veneering material to gold alloy with varying metal surface preparations. J Prostheth Dent 2001; 86: 315-319.
31) Erdemir U, Sancakli HS, Sancakli E, Eren MM, Ozel S, Yucel T, Yildiz E. Shear bond strength of a new self-adhering flowable composite resin for lithium disilicate-reinforced CAD/CAM ceramic material. J Adv Prosthodont 2014; 6: 434-443.