The effect of subpressure on the bond strength of resin to zirconia ceramic

Yong-Mei Li*, Rui-Shen Zhuge*, Zu-Tai Zhang*, Yue-Ming Tian, Ning Ding
School of Stomatology, Capital Medical University, Beijing, China

☯ These authors contributed equally to this work.
+ zutaizhang@ccmu.edu.cn

Abstract

Objective
This study was conducted to investigate the effect of subpressure on the bond strength of resin to zirconia ceramic. The subpressure would create a pressure gradient which could clean out the bubbles in the adhesives or bonding interface.

Methods
Twenty-eight pre-sintered zirconia discs were fabricated. Half of them were polished (group P, n = 14), and the rest were sandblasted (group S, n = 14). After sintered, the surface roughness of the zirconia discs was measured. Then, they were randomly divided into two subgroups (n = 7). The groups were named as follows: PC: P + no additional treatments; PP: P + 0.04 MPa after application of adhesives; SC: S + no additional treatments; and SP: S + 0.04 MPa after application of adhesives. Resin columns were bonded to the zirconia specimens to determine shear bond strength (SBS). The bonding interfaces were observed and the fracture modes were evaluated. Statistical analysis was performed on all data.

Results
The surface roughness of group S was significantly higher than that of group P (P<0.05). The SBS values were PC = 13.48 ± 0.7 MPa, PP = 15.22 ± 0.8 MPa, SC = 17.23 ± 0.7 MPa and SP = 21.68 ± 1.4 MPa. There were significant differences among the groups (P<0.05). Scanning electron microscopy (SEM) results showed that the adhesives of group SP and PP were closer and denser to the zirconia ceramic than that of group PC and SC. The proportion of the mixed fracture mode significantly increased after adding subpressure (P<0.05).

Conclusion
Subpressure can improve the shear bond strength of resin to zirconia ceramics and increase micro-infiltration between the adhesives and the zirconia ceramics, especially on the rough surfaces.
1. Introduction

With the increasing demand for esthetics in prosthodontic treatments, various all-ceramic restorations have been widely used in clinical settings [1]. Zirconia demonstrates superior mechanical properties, chemical stability, biocompatibility and tooth-like color [2] compared with other ceramic materials, making it a commonly used material in dentistry for all-ceramic crowns [3], posts [4], orthodontic brackets [5, 6] and even implants [7, 8]. However, the conventional cementation techniques do not provide an adequate level of bond strength for the zirconia [9]. This is because etching with hydrofluoric acid and silanization had no positive effects on the zirconia bonding due to its resistance to acids and the absence of silicon oxide [10].

Many surface treatments have been used to improve the bond strength of resin to zirconia ceramics over the past two decades, including air abrasion [11], diamond burs abrasion [12], silica (tribochemical) coating [13], silicoating [14, 15], selective infiltration etching [16] and laser [17, 18]. However, many studies have found microscopic cracks because these surface treatments can accelerate tetragonal-to-monoclinic (t→m) phase transformation [19–22], and other researchers found that airborne particle abrasion decreased micro tensile bond strength [23]. Additionally, the addition of coatings can result in poor adhesion to the zirconia [24]. Previous research has suggested that sandblasting before sintering is a useful method for significantly increasing the surface roughness, and it is considered to reverse the transformation (m→t) caused by sandblasting during the sintering process [19, 25]. Additionally, Nobutaka et al. [26] focused on the effect of a primer on bonding to the zirconia. Wei et al. [27] suggested that a high pressure blowing method could improve the adhesion of resin to dentin.

The subpressure infiltration technique is based on the vacuum infiltration theory [28, 29], which is a simple and one-step technique to close micro-voids, repair material defects and develop various kinds of biomaterial scaffolds [30]. The purpose of the current study was to evaluate the effect of a novel subpressure infiltration technique on the bonding of resin to zirconia.

2. Materials & methods

Fig 1 shows the flow chart of the experiment. The process in detail was as follows.

2.1 Preparation of the zirconia discs

Twenty-eight discs (16 mm in diameter, 1.5 mm in thickness) were fabricated from pre-sintered yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic blocks (High transparent material, Nissin-Metec China Co., Ltd., China) with a diamond saw (Isomet 4000 Linear Precision Saw, Buehler, USA) under copious water. Half of the discs were polished (P) using 1200 grit waterproof silicone-carbide paper, the other half were sandblasted (S) (TJK-SP II, Tianjin Haide, China) using 120 μm Al2O3 particles at 2 bar blasting pressure from a distance of 10 mm for 10 sec. All zirconia discs were densely sintered (Kawo Everest Therm, Germany) at a heating speed of 5˚C/min to 1450˚C, holding for 2 h, and then naturally cooling to room temperature. All zirconia discs were ultrasonically cleansed in deionized water for 10 min to remove surface debris prior to use, dried with clean air flow and kept in a desiccator.

2.2 Surface roughness

The surface roughness of the zirconia ceramic was measured after being densely sintered with a surface roughness measuring instrument (JB-4C, Shanghai Taiming Optical Instrument Co.,
Ltd., China), and the zirconia ceramic surfaces were observed by 3-D roughness reconstruction with scanning electron microscopy (Phenom-world Co., LTD., Netherlands, SEM).

2.3 Subpressure infiltration technique

In this study, the novel subpressure device consists of a subpressure box, a vacuum pump, a vacuum gauge, and a three-way valve. The schematic diagram of the subpressure infiltration technique is shown in Fig 2. The specimens were put in the subpressure box through the door. The
vacuum gauge can adjust the pressure in the subpressure box, and by turning the three-way valve outside, the inner pressure in the subpressure box could return to atmospheric pressure.

2.4 Specimen of resin to zirconia

Prior to the application of subpressure, group P (polished) and S (sandblasted) were randomly divided into two subgroups, respectively, labeled PC: P + no additional treatments, PP: P + 0.04 MPa after applying adhesives, SC: S + no additional treatments, and SP: S + treated with 0.04 MPa after applying adhesives.

A piece of transparent tape (20 μm in thickness) with a circular hole (4 mm in diameter) was placed on the zirconia surface to define the bonding area. The primer and luting agents (Variolink N, Ivoclar Vivadent AG, Liechtenstein) were applied to the zirconia surface according to the manufacturer's instructions. Group PP and SP were placed in the subpressure box under 0.04 MPa for 3 min after applying the primer and luting agents, respectively (For the mechanism, see preceding 2.3). The resin column (5 mm in diameter, 4 mm in thickness) was made of composite resin (Valux™ Plus, 3M ESPE, USA) with a stainless steel mold in advance. Then, the resin column was put on the zirconia surface, and covered over the hole of transparent tape exactly. 20 N of force was loaded on the resin column for 1 min before curing from three directions for 10 sec with a light curing unit (Elipar™ 2500, 3M ESPE, USA) and holding for 3 min. All specimens were stored in distilled water at 37°C for 24 hours. Finally, zirconia substrates of each specimen were embedded in methyl methacrylate resin.

2.5 Bonding interface observation

One specimen was randomly selected from each group and sectioned perpendicular to the bonding surface to observe the bonding interface by SEM.

2.6 Shear bond strength test

Shear bond strength (SBS) test was performed using a universal testing machine (AG-X Plus, Shimadzu Co., LTD., Shimadzu, Japan) at a cross-head speed of 0.5 mm/min until the resin column was separated from the zirconia substrate. The SBS values were calculated according to the following formula:

\[
SBS = \frac{F_{\text{max}}}{S}
\]

where SBS is shear bond strength (MPa), \(F_{\text{max}}\) is maximum load (N), and \(S\) is the cross-section surface area (mm²).

2.7 Fractured mode analysis

The debonded surfaces of the specimens were inspected by SEM (500× magnification) to assess the fracture modes, which were classified as the following:

1. Cohesive fracture: A fracture within the resin layer, adhesives or zirconia.
2. Interface fracture: A fracture at the adhesive-zirconia or resin-adhesive interface.
3. Mixed fracture: Both cohesive and interface fractures were observed in the same disc.

2.8 Statistical analysis

Student's t-test was performed to evaluate the differences in roughness between group P and S. Two-way ANOVA was performed to study the contributions of the subpressure, the sandblast
and the interaction of these factors on the SBS. A chi-square test was used to examine the difference in the distribution of the fracture modes (SPSS 19.0 for Windows, SPSS Inc., USA). The statistical significance level was set at $\alpha = 0.05$.

3. Results

3.1 Surface roughness

The surface roughness values of group P and S were $0.92 \pm 0.81 \mu m$ and $3.13 \pm 1.01 \mu m$, respectively, and there was a significant difference between them ($P<0.05$). Representative 3-D images are shown in Fig 3. Group S showed more irregular outlines compared with group P in which the surface was more planar.

3.2 Bonding interface observation

Fig 4 shows the bonding interfaces of the resin to the zirconia. Compared with group P, the zirconia surface of group S was wavier and more uneven. There was an obvious gap (arrow) in subgroup PC between the adhesives and the zirconia, whereas the bonding interface was uniform and intact with no voids or defects in subgroup PP. The adhesives were able to be added into the micro fissures of the zirconia surface in subgroup SP, while there were some voids (arrow) between the adhesives and the zirconia in subgroup SC.

3.3 Shear bond strength test

The mean values and standard deviations of the SBS values were PC = 13.48 ± 0.7 MPa, PP = 15.22 ± 0.8 MPa, SC = 17.23 ± 0.7 MPa, and SP = 21.68 ± 1.4 MPa. There was a significant difference in the SBS among the groups ($P<0.05$) (Fig 5). Two-way ANOVA test showed that both subpressure and sandblast had significant effects to the SBS of resin to zirconia ceramics, and there were significant interaction between them (Table 1).

3.4 Fracture mode analysis

Table 2 shows the distribution of the fracture modes after the SBS test. No cohesive fracture was observed in any group. The fracture mode distribution of group PP and SC was the same and was significantly different compared with group SP and PC ($P<0.05$). Mixed fractures had the highest frequency in group SP, while the group PC showed the lowest percentage of mixed fractures. These groups were significantly different ($P<0.05$).

As Fig 6 shows, there were few adhesives left on the surface of the zirconia in group PC. In group PP, more adhesives could be observed on the zirconia surface. In group SC, there were
still some scattered vacancies (arrow) without adhesives filling them. In group SP, most adhesives remained on the zirconia surface even in the micro pits and micro fissures. Additionally, some resin left on the zirconia surface was visible to the naked eye.

4. Discussion

At present, there are amounts of cases where the retention of restorations is reliant on bonding. Poor bonding may result in restorations becoming loose or dislodged, or it may produce micro-leakage, resulting in plaque accumulation, which can lead to secondary caries, marginal gingivitis, etc. [31]. Hence, the quality of the bonding is of increasing importance and is a dominant factor required for long-term success, especially in regard to acid resistance and silica-free zirconia ceramic restorations. Although many dental techniques [32] have been extensively investigated to improve the bond strength of resin to zirconia ceramics, there is no established protocol that can produce a stable bond which can be easily used at the present time. To achieve higher retention, prevent micro-leakage, and increase the fracture and fatigue resistance of restorations, many studies are under way [13, 26, 27]. However, the subpressure infiltration technique has not been applied in the dental adhesion.

Previous studies have suggested that roughness affects the bond strength [17, 33, 34]. The purpose of this study was to investigate the effect of subpressure on bonding to different rough surfaces. The surface roughness of group S was significantly higher than that of group...
In addition, the SBS values of group S was significantly higher due to the sandblasting (P < 0.05), which was in agreement with the findings of other researchers [19, 33, 34]. In this study, the subpressure infiltration technique was as follows. First, the zirconia surface was covered with adhesives. When the coated zirconia specimens were held in the subpressure chamber, the bubbles between the zirconia and the adhesives or in the adhesives were exhausted due to the pressure gradient. When recovering to atmospheric pressure, the pressure on the adhesives pushed them into the voids or fissures of the zirconia surface, which made the adhesives close to the zirconia substrates.

Table 1. Test of Two-way ANOVA.

| Source                        | Type III Sum of Squares | df | Mean Square | F     | Sig.  |
|-------------------------------|-------------------------|----|-------------|-------|-------|
| Corrected Model               | 224.644                 | 3  | 74.881      | 83.19 | 0     |
| Intercept                     | 6855.316                | 1  | 6855.316    | 7615.932 | 0    |
| subpressure                   | 57.289                  | 1  | 57.289      | 63.645 | 0     |
| sandblast                     | 156.366                 | 1  | 156.366     | 173.715 | 0    |
| subpressure * sandblast       | 10.989                  | 1  | 10.989      | 12.208 | 0.002 |
| Error                         | 18.003                  | 20 | 0.9         |       |       |
| Total                         | 7097.962                | 24 |             |       |       |
| Corrected Total               | 242.646                 | 23 |             |       |       |

R Squared = .926(Adjusted R Squared = .915)

https://doi.org/10.1371/journal.pone.0179668.t001
As the subpressure infiltration technique mechanism mentioned, SEM results (Fig 4) verified that the adhesives were in closer contact with the zirconia in the subpressure groups (SP and PP); whereas space still remained on the surface of the non-subpressure zirconia specimens, especially on the rougher surface. The SBS of the subpressure group was higher than that of the non-subpressure group, confirming that the subpressure had a significant effect on the bonding. The distribution of the mixed fracture mode was significantly different ($P < 0.05$) under subpressure and sandblasting, which was in agreement with the results of the SBS in this study.

The current theories of mechanics suggest that the adhesive must penetrate into the voids within the adherent surface, and the air bubbles on the interface should be cleared out as possible to promote adhesion. The subpressure could effectively remove air on the interface and

| Fracture mode     | PC  | PP  | SC  | SP  |
|-------------------|-----|-----|-----|-----|
| Cohesive fracture | 0   | 0   | 0   | 0   |
| Interface fracture| 83.3% (5) | 50% (3) | 50% (3) | 33.3% (2) |
| Mixed fracture    | 16.7% (1) | 50% (3) | 50% (3) | 66.7% (4) |

The chi-square analysis revealed that there was a significant difference in the fracture mode between groups ($P < 0.05$) except between group PP and SC ($P > 0.05$).

As the subpressure infiltration technique mechanism mentioned, SEM results (Fig 4) verified that the adhesives were in closer contact with the zirconia in the subpressure groups (SP and PP); whereas space still remained on the surface of the non-subpressure zirconia specimens, especially on the rougher surface. The SBS of the subpressure group was higher than that of the non-subpressure group, confirming that the subpressure had a significant effect on the bonding. The distribution of the mixed fracture mode was significantly different ($P < 0.05$) under subpressure and sandblasting, which was in agreement with the results of the SBS in this study.

The current theories of mechanics suggest that the adhesive must penetrate into the voids within the adherent surface, and the air bubbles on the interface should be cleared out as possible to promote adhesion. The subpressure could effectively remove air on the interface and

![Fig 6. Representative fracture modes of all groups (500 ×).](https://doi.org/10.1371/journal.pone.0179668.g006)
amplify the penetration of the adhesives into the micro-pits and micro-hollows of the zirconia surface, which would consequently increase the bonding area and the mechanical locking function [35, 36]. In addition, the close combination of the adhesives to the zirconia increased the intermolecular forces, and the subpressure infiltration technique could potentially reduce microleakage and improve the bonding property [37, 38].

Furthermore, the viscosity of the adhesives materials, the solvent in the primer or the luting agent could affect the infiltration of the zirconia. Low viscosity of the adhesives is conducive and will allow faster infiltration of the gap. Usually, the strength of low-viscosity adhesives is lower than that of high-viscosity adhesives [39, 40]. Additionally, primers or luting agents containing functional monomers could increase the chemical bonding and wettability [26,41]. The configuration of the zirconia surface could affect the infiltration as well, and the subpressure value and action time could also affect the infiltration capacity. Further studies are being conducted.

Finally, compared to other treatments on the zirconia, the subpressure infiltration technique had the advantages of lower technical sensitivity, low cost, short processes and a wide application to other materials. Additionally, the subpressure infiltration technique was not limited to the shape of the restorations.

5. Conclusions
This in vitro study suggests that the subpressure is an efficient method of improving the bond strength of resin to zirconia, and it is more effective on a rough surface.

Supporting information
S1 Text. Raw data for SBS.
(DOCX)
S1 Fig. The physical map of subpressure box.
(TIF)

Author Contributions
Conceptualization: YML RSZ ZTZ YMT.
Data curation: YML RSZ.
Formal analysis: YML RSZ ZTZ YMT.
Investigation: YML RSZ.
Methodology: YML RSZ ZTZ YMT.
Project administration: ZTZ.
Resources: ZTZ ND.
Writing – original draft: YML.
Writing – review & editing: YML RSZ ZTZ YMT.

References
1. Hata U, Kawauchi D, Yamamura O, Fujiwara S. Chromatic Study of All-ceramic Restorations: Relative Translucency of 9 All-ceramic Core Materials. Prostodontic Research & Practice. 2008; 7(2): 159–155.
2. Nordahl N, Vult vS P, Larsson C. Fracture strength of ceramic monolithic crown systems of different thickness. Journal of Oral Science. 2015; 57(3):255–261. https://doi.org/10.2334/josnusd.57.255 PMID: 26369491

3. Vult vS P, Ebbesson S, Holmgren J, Nilner K. Fracture strength of two oxide ceramic crown systems after cyclic pre-loading and thermocycling. Journal of Oral Rehabilitation. 2006; 33(9):682. https://doi.org/10.1111/j.1365-2842.2005.01604.x PMID: 16922742

4. Ahmad I. Zirconium oxide post and core system for the restoration of an endodontically treated incisor. Practical Periodontics & Aesthetic Dentistry Ppad. 1999; 11(2):197–204; quiz 206.

5. Keith O, Kusy RP, Whitley JQ. Zirconia brackets: An evaluation of morphology and coefficients of friction. American Journal of Orthodontics & Dentofacial Orthopedics. 1994; 106(6):605–14.

6. Landsiedel R, Fabian E, Lan MH, Wohleben W, Wiench K, Oesch F, et al. Material of orthodontic brackets. Archives of Toxicology. 2012; 86(7):1021–60.

7. Yildirim M, Edelhoff D, Hanisch O, Spiekermann H. Ceramic abutments—a new era in achieving optimal esthetics in implant dentistry. International Journal of Periodontics & Restorative Dentistry. 2000; 20(1):81–91.

8. Inokoshi M, De MJ, Minakuchi S, Van MB. Meta-analysis of bonding effectiveness to zirconia ceramics. Journal of Dental Research. 2014; 93(4):329–34. https://doi.org/10.1177/0022034514524228 PMID: 24563487

9. Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. Quintessence International. 2007; 38(9):745–753. PMID: 17873981

10. Dérand P, Dérand T. Bond strength of luting cements to zirconium oxide ceramics. International Journal of Prosthodontics. 2000; 13(2):131–135. PMID: 11203621

11. Yang B, Barlovi A, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. Dental Materials. 2009; 26(1):44–50.

12. Michida SM, Kimpfura ET, Dos SC, Souza RO, Bottino MA, Özcan M. Effect of air-abrasion regimens and fine diamond bur grinding on flexural strength, Weibull modulus and phase transformation of zirconium dioxide. Journal of Applied Biomaterials & Fundamental Materials. 2015; 13(3).

13. Moezzizadeh M, Nojedehi an H, Haghi HV. Effect of bioglass and silica coating of zirconia substrate on its bond strength to resin cement. Dental Materials Journal. 2017; 36(1):54–62. https://doi.org/10.4012/dmj.2015-382 PMID: 27928098

14. Goracci C, Ferrari M. Current perspectives on post systems: a literature review. Australian Dental Journal. 2011; 56 (Suppl 1):77–83.

15. Kasraei S, Rezaeisoufi L, Heidari B, Vafaee F. Bond strength of resin cement to CO₂ and Er: YAG laser-treated zirconia ceramic. Restorative Dentistry & Endodontics. 2014; 39(4):296–302.

16. Abousheilib MN, Kleverlaan CJ, Feilzer AJ. Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials. Journal of Prosthetic Dentistry. 2007; 98(5):379.. https://doi.org/10.1016/S0022-3913(07)60129-1 PMID: 18021827

17. Paranhos MP, Luiz Henrique B, Pascal M. Effect of Nd: YAG laser and CO₂ laser treatment on the resin bond strength to zirconia ceramic. Quintessence International. 2011; 42(42):79–89.

18. Cavalcanti AN, Pilecki P, Foxton RM, Watson TF, Oliveira M, Gianinni M, et al. Evaluation of the surface roughness and morphologic features of Y-TZP ceramics after different surface treatments. Photomedicine & Laser Surgery. 2009; 27(3):473.

19. He M, Zhang Z, Zheng D, Ding N, Liu Y. Effect of sandblasting on surface roughness of zirconia-based ceramics and shear bond strength of veneering porcelain. Dental Materials Journal. 2014; 33(6):778–785. PMID: 25311341

20. Wei Y, Zhang Z, Ding N, Zheng D, Li H, Liu X. Improvement in Ti-porcelain bonding by SiO₂ modification of titanium surface through cast method. Dental Materials Journal. 2013; 32(4):564–571. PMID: 23903637

21. Fischer J, Stawarczyk B, Sailer I, Hämmerle CH. Shear bond strength between veneering ceramics and ceria-stabilized zirconia/alumina. Journal of Prosthetic Dentistry. 2010; 103(5):267–274. https://doi.org/10.1016/S0022-3913(10)60056-X PMID: 20416409

22. Saito A, Komine F, Blatz MB, Matsumura H. A comparison of bond strength of layered veneering porcelains to zirconia and metal. Journal of Prosthetic Dentistry. 2010; 104(4):247–57. https://doi.org/10.1016/S0022-3913(10)60133-3 PMID: 20875529

23. Harding AB, Norling BK, Teixeira EC. The Effect of Surface Treatment of the Interfacial Surface on Fatigue-Related Microtensile Bond Strength of Milled Zirconia to Veneering Porcelain. Journal of Prosthodontics-implant Esthetic & Reconstructive Dentistry. 2012; 21(5):346–352.
24. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: Where are we now? Dental Materials. 2011; 27(1):71–82. https://doi.org/10.1016/j.dental.2010.10.022 PMID: 21094526

25. Moon JE, Kim SH, Lee JB, Ha SR, Choi YS. The effect of preparation order on the crystal structure of yttria-stabilized tetragonal zirconia polycrystal and the shear bond strength of dental resin cements. Dental Materials. 2011; 27(7):651–63. https://doi.org/10.1016/j.dental.2011.03.005 PMID: 21351018

26. Nobutaka A, Koizumi H, Nogawa H, Nakayama D, Kodaira A, Matsumura H. Effect of mechanochemical surface preparation on bonding to zirconia of a tri-n-butylborane initiated resin. Dental Materials Journal. 2016; 36: 19–26. https://doi.org/10.4012/dmj.2016-099 PMID: 27928101

27. Wei Z, Niu LN, Li H, Huang L, Fang M, Chang G, et al. Improved secondary caries resistance via augmented pressure displacement of antibacterial adhesive. Scientific Reports. 2016; 6:22269. https://doi.org/10.1038/srep22269 PMID: 26928742

28. Martin J, Mijangos C. Tailored Polymer-Based Nanofibers and Nanotubes by Means of Different Infiltration Methods into Alumina Nanopores. Langmuir the Acs Journal of Surfaces & Colloids. 2009; 25(2): 1181–7.

29. Vogt U, Gorbar M, Eggenschwiler PD, Colombo P. Improving the properties of ceramic foams by a vacuum infiltration process. Journal of the European Ceramic Society. 2010; 30(15):3005–3011.

30. Golafshan N, Gharibi H, Karazinha M, Fathi M. A facile one-step strategy for development of a double network fibrous scaffold for nerve tissue engineering. Biofabrication. 2017; 9(2):025008 https://doi.org/10.1088/1758-5090/aa68ed PMID: 28452328

31. Rashid H. The effect of surface roughness on ceramics used in dentistry: A review of literature. European Journal of Dentistry. 2014; 8(4):571–9. https://doi.org/10.4103/1305-7456.143646 PMID: 25512743

32. Pozzobon JL, Wandscher VF, Rippe MP, Valandro LF. Influence of zirconia surface treatments on resin cement bonding and phase transformation. Journal of Adhesion Science and Technology. 2017; 19(1): 7–19.

33. Akin GE, Kaval ME, Turk T, Akin H. Surface roughness and bond strength of zirconia posts to a resin cement after various surface pretreatments. Photomedicine & Laser Surgery. 2015; 33(5):246.

34. Budhe S, Ghumatkar A, Birajdar N, Banea MD. Effect of surface roughness using different adherend materials on the adhesive bond strength. Applied Adhesion Science. 2015; 3(1):20.

35. Kitayama S, Nikaido T, Takahashi R, Zhu L, Ikeda M, Foxton RM, et al. Effect of primer treatment on bonding of resin cements to zirconia ceramic. Dental Materials. 2010; 26(5):426–432. https://doi.org/10.1016/j.dental.2009.11.159 PMID: 20102781

36. Kadir MRA, Osman K, Zainuddin AI. The Use of Mechanical Interlock in Dental Prosthesis. Springer Berlin Heidelberg. 2008; 21(1):457–460.

37. Manicone PF, Rossi IP, Raffaelli L. An overview of zirconia ceramics: basic properties and clinical applications. Journal of Dentistry. 2007; 35(11):819–826. https://doi.org/10.1016/j.jdent.2007.07.008 PMID: 17825465

38. Ozcan M, Nijhuis H, Valandro LF. Effect of Various Surface Conditioning Methods on the Adhesion of Dual-cure Resin Cement with MDP Functional Monomer to Zirconia after Thermal Aging. Dental Materials Journal. 2008; 27(1):99–104. PMID: 18309618

39. Hwang DS, Zeng H, Srivastava A, Srivastava A, Krogstad DV, Tirrell M, Israelachvili JN, et al. Viscosity and interfacial properties in a mussel-inspired adhesive coacervate. Soft Matter. 2010; 6(14):3232. https://doi.org/10.1039/C002632H PMID: 21544267

40. Montes MA, Goes MFD, Cunha MR, Soares AB. A morphological and tensile bond strength evaluation of an unfilled adhesive with low-viscosity composites and a filled adhesive in one and two coats. Journal of Dentistry. 2001; 29(29):435–441.

41. Bulbul M, Kesim B. The effect of primers on shear bond strength of acrylic resins to different types of metals. Journal of Prosthetic Dent. 2010; 103: 303–308.