



HOKKAIDO UNIVERSITY

Title	Durability for mechanical property and bonding performance of resin materials bonded to CAD/CAM resin block
Author(s)	張, 紅
Degree Grantor	北海道大学
Degree Name	博士(歯学)
Dissertation Number	甲第15948号
Issue Date	2024-03-25
DOI	https://doi.org/10.14943/doctoral.k15948
Doc URL	https://hdl.handle.net/2115/94533
Type	doctoral thesis
File Information	Hong_Zhang.pdf



博 士 論 文

Durability for mechanical property and bonding performance of resin materials bonded to CAD/CAM resin block

CAD/CAM レジンブロックに対するレジン系材料の
機械的性質および接着力における耐久性について

令和 5 年 3 月 申請

北海道大学
大学院歯学院口腔医学専攻

張 紅

Introduction

Various congenital or acquired factors, such as dental fluorosis, tetracycline staining, enamel hypoplasia, twisted teeth, malformed teeth, small teeth, and dental caries, can lead to abnormal tooth shape or color, greatly affecting the esthetics, occlusal dysfunctions and even healthy dental tissue of patients, requiring restorative treatment [1]. With the improvement of living standards and esthetic requirements, esthetic restoration of anterior teeth not only requires restoring the tooth's shape, color, and function but also aims to preserve and protect as much healthy dental tissue as possible. Therefore, laminate veneers become the preferred choice for the restoration of anterior teeth [2].

Laminate veneers, as indirect restorations, cover the tooth surface by adhesive technology with different materials. They restore the teeth's shape and improve their color, function and healthy tissue [3]. Based on different materials, laminate veneers are mainly divided into porcelain veneers and resin veneers. Porcelain veneers are highly accepted by patients for their rather good chemical stability and biocompatibility [6]. However, their high brittleness, high difficulty of processing, and considerable repair challenges limit their widespread clinical application [7]. With the development of CAD/CAM technology and the development of new cuttable resin blocks, indirect resin laminate veneers are becoming more and more popular in clinic for their advantages such as higher fracture resistance and fatigue strength, easier processing and repair, better marginal adaptation, easier removal, and lower cost [8, 9]. However, they are detrimental to adhesion because they are highly polymerized and free of residual monomers [10].

Bonding is a critical step to ensure the retention and marginal closure of laminate

veneers which affects the clinical service life of restorations [11]. Improper bonding process can lead to structural defects at the bonding interface, compromising interface stability, which may result in veneer debonding, fracturing, and even restoration failure [12]. Resin cements are commonly used for laminate veneers including light-cured and dual-cured resin cements [13]. Dual-cured resin cements combine the light-activated polymerization with the chemical curing mode, which can compensate the curing light [14]. The light-initiated component can be light-cured immediately, while redox-initiated component cures gradually [15]. It was also reported that the bond strength of dual-cured resin cement was higher than that of light-cured resin cement [16]. However, it was noticeable that acidic monomers in self-etch adhesives inhibited the polymerization of dual-cured composite resins [17]. Furthermore, the dual-cure resin cements had considerably greater differences in the color change because of the amine [18]. Besides, since light-cured resin cements can only be polymerized when light cured, they can provide sufficient working time and facilitate excess cement removal [19]. After light curing, they can acquire immediate final polymerization and strength [20]. Moreover, light cured resin cements offer better color stability as they do not contain chemical amine initiators [21]. Therefore, light-cured resin cements are usually suggested to bonding thin and translucent laminate veneers. In addition, resin composite was suggested to be used as cement, as its large filler size and big volume, can offer better bond strength and mechanic performance [22].

This purpose of this study was to evaluate the microtensile bond strength (μ TBS) of four different resin materials bonded between dentin and CAD/CAM resin block, as long as their Knoop hardness number (KHN) and cohesive strength. The null hypothesis of the study was that (i) there were no differences in μ TBS, KHN and cohesive strength

of four resin materials, (ii) time do not affect μ TBS, KHN and cohesive strength of four resin materials.

Materials and Methods

This study was approved by Hokkaido University Research Ethics Committee (approval number 2018-9). Twenty-four extracted caries-free human third molars were stored in 0.5% Chloramine-T solution at 4°C and utilized within 6 months after extraction.

1. Materials Used in the Study

Three types of resin cements and one resin composite were used in this study as follows: (1) Panavia Veneer LC (PL), (2) Variolink Esthetic LC (VE), (3) Panavia V5 (PV), and (4) Clearfil AP-X (AP). According to the manufacturer's instructions, these four materials were applied after Panavia V5 tooth primer (PV-Primer), Adhese Universal (AU), and Clearfil SE Bond 2 (SE2). Additionally, the ceramic primers, Clearfil Ceramic Primer Plus, and Monobond Plus, were also used. All materials used in this study, along with their compositions, are shown in Table 1.

2. μ TBS test and fracture mode analysis

The schematic diagram of this procedure is presented in Figure 1. Twenty-four caries-free human third molars and CAD/ CAM (12 sizes; shade A3LT) resin blocks (Katana Avencia Block 2, Kuraray Noritake Dental, Tokyo, Japan) were applied to prepare the specimens. The CAD/CAM resin blocks were sliced into 1.5 mm thickness using a slow-speed diamond saw (Isomet Buehler, IL, USA). The surfaces of resin slices were polished under running water for 60 s with 600-grit SiC paper (Sankyo-Rikagaku Co., Ltd., Tokyo, Japan), sandblasted with 50 μ m alumina powder at 0.2 MPa,

ultrasonically cleaned for 2 min, and then dried with a syringe air blow for 10 s. All CAD/CAM resin slices were etched with phosphoric acid for 5 s, and then cleaned with running water for 20 s, dried for 10 s, and treated with silane coupling agent according to the manufacturer's instructions.

The flat, occlusal middle dentin surface was exposed using a gypsum model trimmer under water cooling and then polished with #600 SiC paper (Sankyo-Rikagaku Co., Saitama, Japan) for 60 s under running water to produce a standardized smear layer. All teeth were randomly divided into four groups based on the types of resin materials: PL, VE, PV, and AP. For the PL and PV groups, PV-Primer was applied to the exposed dentin surface following the manufacturer's recommendation. In the VE group, AU was applied to the exposed dentin surface. The AP group received the application of primer and bonding agent SE2 on the exposed dentin surface. After applying the adhesive agent, each dentin surface was light-cured for 10 s at 2000 mW/cm² using a cordless LED (Pencure2000, Morita, Tokyo, Japan). Subsequently, resin materials were applied to the tooth surface, and the prepared CAD/CAM resin slices were firmly pressed over the resin materials. The samples were light-cured for 20 s. To obtain the 4 mm standard height for the μ TBS test, the upper side of the CAD/CAM resin slice was applied with Clearfil Ceramic Primer Plus (Kuraray Noritake Dental, Tokyo, Japan) and SE Bond primer, followed by SE Bond bond and light-cured for 10 s. Then, a 2.5 mm height of light-cured resin composite layer was built on the surface of the resin slice. All specimens were stored in distilled water at 37°C for 24 hours.

After 24 hours of storage in distilled water at 37°C, each specimen was longitudinally cross-sectioned with a low-speed diamond saw (IsoMet 1000, Buehler; Lake Bluff, IL, USA) to produce resin-dentin beams (1 ± 0.05 mm²). The beams from

three specimens in each group were stored in distilled water for 6 months and the beams from the other three specimens were tested for μ TBS immediately. Five central beams were selected from each specimen for the μ TBS test (n=15). The beams were fixed to a Ciucchi's jig with cyanoacrylate glue (Dentsply, Otahara, Japan) and subjected to tensile forces at a crosshead speed of 1 mm/min in a desktop testing apparatus (EZ-S, Shimadzu, Kyoto, Japan) until failure occurred. Bond strength was expressed in MPa. The mean bond strength was calculated from fifteen beams obtained from three teeth, resulting in 15 values for each test group.

After testing, the fractured beams were carefully removed from the jigs and mounted on the aluminum stubs. The fracture modes of the specimens were visualized by light microscope ($\times 20$ magnification; Magnifier Light, Asone, Osaka, Japan), then coated with Pt-Pd for 120 s and confirmed by scanning electron microscopy (SEM) (S-4000, Hitachi, Tokyo, Japan) at an accelerating voltage of 10 kV. Failure modes on the dentin sides of beams were taken into consideration and classified as follows: fracture failure in adhesive; fracture failure in cement; fracture failure in dentin; mixed failure in cement and resin block; fracture failure in resin composite.

3. Microhardness measurement

A transparent silicon model was fabricated with a 2 mm height and 10 mm diameter hole. Two glass microscope slides were placed over the top and the bottom of hole to keep the surface smooth. The resin materials were dispensed directly into the hole and light-cured for 20 s both on top and bottom of specimens using a cordless LED at 2000 mW/cm². During the light activation procedure, the light guide was positioned in the middle of the specimen. All procedures were conducted at $23 \pm 2^\circ\text{C}$ temperature and $50 \pm 10\%$ humidity to prevent photo-initiator sensitization. Additionally, a filter was

employed to maintain ambient red light. Ten specimens were prepared for each group. Half of the specimens were tested after 24 hours while others were tested after 6 months of storage in a dry, dark environment at 37°C.

Indentations and microhardness measurements (KHN) were sequentially performed using a microhardness tester with a 20× objective lens and a 25 gf load for 15s. Each specimen was indented for ten times at 0.5 mm intervals, starting from the center and proceeding outward. The long-axis length of each indentation was measured and recorded. Subsequently, the KHN of the specimens was calculated.

4.Cohesive strength test

The resin material was dispensed directly to a transparent silicon model with 20 mm length and 2 mm inner diameter. After light-cured for 20 s at 2000 mW/cm², the specimens were trimmed to the dumbbell shape with a neck diameter of 1 mm, and then stored in the distilled water at 37°C. Twelve specimens were prepared for each group. After 24 hours or 6 months of water storage, the specimens underwent μ TBS were tested to assess cohesive strength, following the previously mentioned test procedure (n=6).

5.Statistical Analysis

Statistical analysis was conducted using the SPSS software package, Statistical Package for the Social Sciences for Windows (SPSS version 25; IBM Corp, Armonk, NY, USA). The normal distribution was assessed using the Shapiro-Wilk test, and the homogeneity of variance was evaluated using Levene's test. Based on the results of these tests, the μ TBS data, KHN, and cohesive strength data were subjected to a two-way ANOVA followed by Tukey HSD post-hoc test ($\alpha=0.05$). What's more, a one-way ANOVA and Tukey HSD post-hoc test were separately conducted to compare

all groups of μ TBS, KHN, and cohesive strength ($\alpha=0.05$).

Result

1. μ TBS

The means and standard deviations of μ TBS values are presented in Table 2. Two-way ANOVA revealed significant effect of materials ($p<0.001$) on the μ TBS, while there were no significant effect of time ($p=0.281$) on the μ TBS. Furthermore, the interaction between materials and time also demonstrated the significance ($p<0.001$).

One-way ANOVA indicated a significant difference among all eight groups ($p<0.001$). The Tukey HSD post-hoc test showed that the μ TBS of the VE group was significantly lower than that of the other three materials both after 24 hours and 6 months ($p<0.001$). There was no significant difference between PL and AP for either the 24 hour or 6 month ($p>0.05$). Moreover, the μ TBS of the PV group significantly increased after 6 months ($p<0.001$).

2. Fracture mode analysis

The failure modes are presented in Table 2, with no observed pretest failures. Figure 2 and 3 show the SEM images of the representative fracture modes at the dentin side at x80 and 1000 magnifications. In the PL, VE, and PV groups, the primary fracture mode was cohesive failure in the resin material, observed both after 24 hours and 6 months. Conversely, in the AP group, the predominant failure mode was cohesive failure in dentin. Notably, there were no instances of adhesive failure or dentin failure in the PL group and no resin cement failure in the PV group for both after 24 hours and 6 months. And the highest percentage of cement failure was observed in the VE group.

3. Micro hardness evaluation

The results of KHN are presented in Table 3. Two-way ANOVA revealed significant effects of materials ($p < 0.001$) and time ($p = 0.017$) on KHN. However, the interaction between materials and time showed no statistical significance. The KHN of AP was significantly higher than that of other materials ($p < 0.001$). The KHN of VE and PL did not show statistical significance ($p = 0.126$), but they were both significantly higher than PV. One-way ANOVA showed a significant difference among all eight groups ($p < 0.001$). The Tukey HSD test indicated that the KHN of all materials showed no significant difference between 24 hours and 6 months ($p > 0.05$).

4. Cohesive strength

The results of cohesive strength are shown in Table 4. Two-way ANOVA revealed a significant effect of materials ($p < 0.001$) on the cohesive strength. However, there was no significant effect of time ($p = 0.841$) on the cohesive strength, and no significant interaction between materials and time ($p = 0.900$). According to the post-hoc test, AP exhibited the highest cohesive strength compared to PL, VE, and PV ($p < 0.001$). Additionally, the cohesive strength of PV was significantly higher than VE and PL ($p < 0.001$), while the cohesive strength of VE and PL had no significant difference ($p = 0.971$).

One-way ANOVA showed a significant difference among all eight groups ($p < 0.001$). The Tukey HSD test indicated that the cohesive strength of all materials showed no significant difference between 24 hours and 6 months ($p > 0.05$).

Discussion

Based on the current results, the first null hypothesis was rejected, which means that μ TBS, microhardness, and cohesive strength were significantly affected by types of

resin materials. The second null hypothesis was partially rejected, symbolizing that the dual-cured resin cement achieved an increasing μ TBS after 6 months of water storage. All materials showed better durability, which means the degradation of all materials didn't excessively occur.

Bond strength is usually to evaluate resin cements [23, 24]. The laboratory methods commonly used for testing bond strength include tensile bond strength test and shear bond strength test [25, 26]. According to the size of the bonding area, these tests can be categorized as macroscopic (bonding area $> 3 \text{ mm}^2$) and microscopic (bonding area $\leq 3 \text{ mm}^2$) [27]. Sano proposed the μ TBS test, which involves testing small areas of tensile bond strength to reduce interference from interface defects during the bonding process and to decrease the likelihood of cohesive failure within the sample [28]. The μ TBS acquires a more concentrated stress distribution and provides a more accurate measure of the precise bonding strength of the adhesives [29]. As a result, they are commonly used in studies on the bond strength of dental bonding materials [30, 31, 32].

In present studies, two-way ANOVA revealed significant effects on resin materials ($p < 0.001$) of the μ TBS, KNH, and cohesive strength. Therefore, the null hypotheses in terms of μ TBS KNH and cohesive strength were all rejected. Meanwhile, there were no significant effects on the durability of all materials.

AP, when bonded with SE2, a two-step, self-etch, light-cure bonding agent, showed very high and durable bond strength. Previous research showed that two-step self-etch adhesives could improve the resin degree of conversion (DC), and produce a high-quality of hybrid layer and acid-base resistant zone at resin-dentin interface [33, 34, 35]. This is possibly due to the presence of 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). It is noticeable that the 10-MDP, the functional monomer, can

dissolve the smear layer, and chemically bond to hydroxyapatite, enhancing monomer permeability [36]. SE2 contributes to the high quality of the hybrid layer and acid-base resistant zone at the resin dentin interface [37]. The light-initiated system may also contribute to the higher μ TBS since it can enhance its conversion degree and mechanical properties [34]. Furthermore, the relatively high amount of inorganic filler and particle size may also be attributed to the higher μ TBS. It has been reported that resin cement with high fillers can significantly enhance the bonding strength and reduce the microleakage of the contact surface between dentin and resin composite [38]. Since AP is a highly filled hybrid-filled composite containing barium glass and colloidal silica fillers with a mean particle size of $3\mu\text{m}$ and a particle distribution between 0.1 mm and 15 mm , with a filler percentage of $70\text{ vol\%}$ ($86\text{ wt\%}$) [39]. Therefore, based on its improved mechanical performance by its higher filler content, resin composite has been suggested to be used as cement [40].

PL, when bonded with Panavia V5 tooth primer, also showed comparable high μ TBS to AP ($p < 0.05$) both after 24 hours and 6 months of water storage. Panavia V5 tooth primer is a self-etching adhesive for tooth structure (dentin and enamel) that seals the tooth-adhesive interface securely as soon as paste is applied to it [41]. This primer can improve surface wettability and its unique touch and cure technology can initiate the polymerization at the adhesive interface without light curing and establish a strong bond to enamel and dentin [36]. The considerably higher μ TBS may also be attributed to its component. According to the manufacturer, the total amount of inorganic filler is approximately, $47\text{ vol\%}$ ($66\text{ wt\%}$) and the particle size of inorganic fillers ranges from $0.05\text{ }\mu\text{m}$ to $8\text{ }\mu\text{m}$ [42]. At the same time, newly developed spherical silica fillers used in PL may further enhance the translucent structure of light-cured resin cement, leading to

improved polymerization kinetics and better bond strength [43], which may also contribute to its good durability.

In this study, the μ TBS of the PV group is significantly lower than AP and PL after 24 hours. However, after 6 months of water storage, it significantly increased, which was comparable to PL and AP. As a dual-cured resin cement, PV contains chemical initiators that continue the polymerization reaction to compensate for incomplete light curing [44]. It was reported that PV could be activated by both photo- and chemical-initiators [45]. Akehashi et al. speculated that PV greatly depends on the chemical initiator [36]. Consequently, PV will provide better bond effect when the restorations are of poor translucency, greater thickness, or darker shade which will impede light curing [46].

Whether after 24 hours or 6 months of water storage, the μ TBS of VE is significantly the lowest. According to the manufacture, the total volume of inorganic fillers is approximately 38 vol % and the mean particle size is 0.1 μ m. The other possible reason may be the universal adhesive. It was argued that universal adhesives mix together components that previously belonged to multiple bottles and do not significantly improve adhesive performance [47].

Another important indicator for evaluating bonding performance is the distribution of fracture modes. The proportion of various fracture modes is often related to the bonding strength of the adhesive material. Generally, when the failure occurs at the bonding interface, the value of the bonding strength can best reflect the bonding property of the adhesive. Van Landuyt found a positive correlation between bond strength and fracture modes [48]. For the PL, VE, and PV groups, the dominant fracture mode was in cement while AP group was in dentin both after 24 hours and 6 months of

water storage.

Surface microhardness is one of the most important parameters for evaluating physical properties of dental materials. It has been reported that resin hardness is strongly related to its degree of conversion (DC), which means the higher DC is, the greater amount of cross-linked polymers are [15]. However, it can also be affected by the cross-linking density in the polymer matrix, the chemical composition, amount of fillers, and matrix composition [49]. Comparing to resin cement, resin composite usually has a higher filler content and filler size [50, 51]. The highest KHN of AP may be related to its filler size and volume. In this research, the KHN of all tested materials were not decreased after 6 months, which suggests that all materials have stable durability. It was reported that DC of dual-cured cements was usually than light-cured cements due to its chemical cure. However, in this study, the KHN of PL was significantly higher than PV, which suggested that the specific spherical silica fillers may get better translucency to improve the DC.

Cohesions are an attraction process that occurs between similar molecules, primarily as the result of chemical bonds that have formed between the individual components of the adhesive or luting agent [52]. Thus, cohesions may be defined as the internal strength of an adhesive. It is common to increase the filler volumes and particle sizes to improve the cohesive strength of the composite materials [52]. It is noteworthy that VE displayed comparable cohesive strength to PL. When the specimen of cohesive strength was prepared, it was under the light directly while the μ TBS was beneath the resin slice. It could be speculated that VE is more sensitive to the curing-light attenuation than PL.

Conclusions

Within the limitations of this in vitro study of resin materials, it can be concluded that:

1. When bonded between dentin and CAD/CAM resin block, PL, PV and AP showed better bond strength and good durability.
2. The KHN and cohesive strength of all resin materials showed significant differences. However, all of them have good durability.
3. PL exhibited good bonding performance and durability, which might be attributed to its specific translucency structure.

Further studies would focus on the color stability, water sorption, and degree of conversion to verify this research results.

Acknowledgments

The authors thank the staff of the Department of Restorative Dentistry and the Department of Dental Medical Laboratory for helping.

Reference

1. Demarco, F.F.; Collares, K.; Coelho-de-Souza, F.H.; Correa, M.B.; Cenci, M.S.; Moraes, R.R.; Opdam, N.J. Anterior composite restorations: A systematic review on long-term survival and reasons for failure. *Dent. Mater.* 2015,31,1214–1224.
2. Calamia, J.R. Etched porcelain veneers: The current state of the art. *Quintessence Int.* 1985, 16, 5–12.
3. Hong N, Yang H, Li J, et al. Effect of Preparation Designs on the Prognosis of Porcelain Laminate Veneers: A Systematic Review and Meta-Analysis[J]. *Oper Dent*, 2017,42(6): E197-E213.

4. F A Spitznagel, J Boldt, P C Gierthmuehlen. CAD/CAM Ceramic Restorative Materials for Natural Teeth. J Dent Res. 2018 ,97(10):1082-1091
5. Zhang Y, Kelly JR. 2017. Dental ceramics for restoration and metal veneering. Dent Clin North Am. 61(4):797–819.
6. Yousra H AlJazairy. Survival Rates for Porcelain Laminate Veneers: A Systematic Review Eur J Dent. 2021 May;15(2):360-368.
7. M M M Gresnigt, M S Cune, K Jansen, et al. Randomized clinical trial on indirect resin composite and ceramic laminate veneers: Up to 10-year findings. J Dent. 2019 Jul;86:102-109.
8. M M M Gresnigt, M S Cune, K Jansen, et al. Randomized clinical trial on indirect resin composite and ceramic laminate veneers: Up to 10-year findings. J Dent. 2019 Jul;86:102-109
9. Lawson NC, Bansal R, Burgess JO. Wear, strength, modulus and hardness of CAD/CAM restorative materials. Dent Mater 2016, 32: e275-e283.
10. Flury S, Schmidt SZ, Peutzfeldt A, Lussi A. Dentin bond strength of two resin ceramic computer-aided design/ computer-aided manufacturing (CAD/CAM) materials and five cements after six months storage. Dent Mater J 2016;35: 728-735.
11. Edson Araujo, Jorge Perdigão. Anterior Veneer Restorations - An Evidence-based Minimal-Intervention Perspective. J Adhes Dent. 2021 Apr 7;23(2):91-110.
12. Alenezi A, Alsweed M, Alsidrani S, et al. Long-Term Survival and Complication Rates of Porcelain Laminate Veneers in Clinical Studies: A Systematic Review. J Clin Med. 2021 Mar 5;10(5):1074.
13. Assaf A, Azer SS, Sfeir A, et al. Risk Factors with Porcelain Laminate Veneers Experienced during Cementation: A Review. Materials (Basel). 2023 Jul

10;16(14):4932.

14. Alovisi M, Scotti N, Comba A, et al. Influence of polymerization time on properties of dual-curing cements in combination with high translucency monolithic zirconia. *J Prosthodont Res*. 2018 Oct;62(4):468-472.

15. De Souza G, Braga RR, Cesar PF, et al. Correlation between clinical performance and degree of conversion of resin cements: a literature review. *J Appl Oral Sci*. 2015 Jul-Aug;23(4):358-68.

16. Novais VR, Raposo LH, Miranda RR, et al. Degree of conversion and bond strength of resin-cements to feldspathic ceramic using different curing modes. *J Appl Oral Sci*. 2017 Jan-Feb;25(1):61-68

17. Haller B. Which self-etch bonding systems are suitable for which clinical indications? *Quintessence Int*. 2013 Oct;44(9):645-61

18. Hardan L, Bourgi R, Hernández-Escamilla T, et al. Color stability of dual-cured and light-cured resin cements: A systematic review and meta-analysis of in vitro studies. *J Prosthodont*. 2023 Aug 31.

19. Gabriel Felipe Bragança, Arthur Silva Vianna, Flávio Domingues Neves, et al. Effect of exposure time and moving the curing light on the degree of conversion and Knoop microhardness of light-cured resin cements. *Dent Mater*. 2020 Nov;36(11):e340-e351.

20. Ghodsi S, Shekarian M, Aghamohseni MM, et al. Resin cement selection for different types of fixed partial coverage restorations: A narrative systematic review. *Clin Exp Dent Res*. 2023 Jul 10.

21. Janes F P, Brunna K A G, Cibele C A K, et al. Color stability of ceramic veneers as a function of resin cement curing mode and shade: 3-year follow-up. *PLoS One*. 2019 Jul 1;14(7):e0219183.

22. Patussi AFC, et al. Preheating of dental composite resins: A scoping review. *J Esthet Restor Dent*. 2023 Jun;35(4):646-656.
23. Santi MR, Lins RBE, Sahadi BO, et al. Evaluation of pretreatments on intra-radicular dentin bond strength of self-adhesive resin cements. *J Esthet Restor Dent*. 2022 Oct;34(7):1051-1059.
24. Nima G, Makishi P, Fronza BM, et al. Polymerization Kinetics, Shrinkage Stress, and Bond Strength to Dentin of Conventional and Self-adhesive Resin Cements. *J Adhes Dent*. 2022 Sep 28;24(1):355-366.
25. Samran A, Al-Ammari A, El Bahra S, et al. Bond strength durability of self-adhesive resin cements to zirconia ceramic: An in vitro study. *J Prosthet Dent*. 2019 Mar;121(3):477-484.
26. Kim AJ, Shin SJ, Yu SH, et al. Shear bond strengths of various resin cements between three types of adherends and bovine teeth with and without thermocycling. *Dent Mater J*. 2022 Apr 1;41(2):323-332.
27. Otani A, Amaral M, May LG, et al. A critical evaluation of bond strength tests for the assessment of bonding to Y-TZP. *Dent Mater*. 2015 Jun;31(6):648-56.
28. Sano H, Shono T, Sonoda H, et al. Relationship between surface area for adhesion and tensile bond strength evaluation of a micro-tensile bond test [J]. *Dent Mater*,1994, 10(4):236-240.
29. Sano H, Chowdhury AFMA, Saikaew P, et al. The microtensile bond strength test: Its historical background and application to bond testing. *Jpn Dent Sci Rev*. 2020;56(1):24-31.
30. Al-Turki L, Merdad Y, Abuhaimeed TA, et al. Repair bond strength of dental computer-aided design/computer-aided manufactured ceramics after different surface

treatments. *J Esthet Restor Dent*. 2020 Oct;32(7):726-733.

31. Song CH, Choi JW, Jeon YC, et al. Comparison of the Microtensile Bond Strength of a Polyetherketoneketone (PEKK) Tooth Post Cemented with Various Surface Treatments and Various Resin Cements. *Materials (Basel)*. 2018 May 29;11(6):916.

32. Shibata S, Vieira LC, Baratieri L, et al. Evaluation of microtensile bond strength of self-etching adhesives on normal and caries-affected dentin. *Dent Mater J*. 2016;35(2):166-73.

33. Chowdhury, A.F.M.A.; Alam, A.; Yamauti, M.; Álvarez-Lloret, P.; Saikaew, P.; Carvalho, R.M.; Sano, H. Characterization of an Experimental Two-Step Self-Etch Adhesive's Bonding Performance and Resin-Dentin Interfacial Properties. *Polymers* 2021, 13, 1009.

34. Sato, K.; Hosaka, K.; Takahashi, M.; Ikeda, M.; Tian, F.; Komada, W.; Nakajima, M.; Foxton, R.; Nishitani, Y.; Pashley, D.H.; et al. Dentin Bonding Durability of Two-step Self-etch Adhesives with Improved Degree of Conversion of Adhesive Resins. *J. Adhes. Dent*. 2017, 19, 31–37.

35. Nikaido T, Nurrohman H, Takagaki T, et al. Nanoleakage in Hybrid Layer and Acid-Base Resistant Zone at the Adhesive/Dentin Interface. *Microsc Microanal*. 2015 Oct;21(5):1271-7.

36. Akehashi S, Takahashi R, Nikaido T, et al. Enhancement of dentin bond strength of resin cement using new resin coating materials. *Dent Mater J*. 2019 Dec 1;38(6):955-962.

37 Guan R, Takagaki T, Matsui N, Sato T, Burrow MF, Palamara J, et al. Dentin bonding performance using Weibull statistics and evaluation of acid-base resistant zone formation of recently introduced adhesives. *Dent Mater J* 2016; 35: 684-693.

38. Barbon FJ, Moraes RR, Isolan CP, et al. Influence of inorganic filler content of resin luting agents and use of adhesive on the performance of bonded ceramic. *J Prosthet Dent.* 2019;122:566-e1.
39. Loomans BA, Cardoso MV, Opdam NJ, et al. Surface roughness of etched composite resin in light of composite repair. *J Dent.* 2011 Jul;39(7):499-505.
40. Magne P, Razaghy M, Carvalho MA, et al. Luting of inlays, onlays, and overlays with preheated restorative composite resin does not prevent seating accuracy. *Int J Esthet Dent.* 2018;13:318–332
41. Garcia IM, Soto-Montero J, Collares FM, et al. Bonding of Resin Cements to Ultra-Translucent Zirconia After Aging for 24 Hours and 1 Year. *Int J Prosthodont.* 2022 Jul-Aug;35(4):460-468.
42. Panavia Veneer LC. Challenging Task, Great Solution. Available online: <https://www.kuraraynoritake.eu/en/panavia-veneer> (accessed on 17 August 2022).
43. Kawamura M, Toida Y, Hoshika S, et al. Influence of Novel Experimental Light-Cured Resin Cement on Microtensile Bond Strength. *Polymers (Basel).* 2022 Sep 28;14(19):4075.
44. Teneshia N, Amar K, Stephen C A, et al. Bond strength of dual-cured resin cement used with dual-cured adhesives. *Gen Dent.* 2020 May-Jun;68(3):72-77.
45. Tsujimoto A, Barkmeier WW, Takamizawa T, Watanabe H, Johnson WW, Latta MA, et al. Simulated localized wear of resin luting cements for universal adhesive systems with different curing mode. *J Oral Sci* 2018; 60: 29-36.
46. Kondo T, Kakinuma H, Fujimura K, et al. Incomplete Polymerization of Dual-Cured Resin Cement Due to Attenuated Light through Zirconia Induces Inflammatory Responses. *Int J Mol Sci.* 2023 Jun 7;24(12):9861.

47. Chen C, Niu LN, Xie H, et al. Bonding of universal adhesives to dentine-Old wine in new bottles? [J]. *Journal of Dentistry*, 2019, 43(5): 525-536.
48. Van Landuyt K L, De Munck J, Snauwaert J, et al. Monomer-Solvent Phase Separation in One-step Self-etch Adhesives. *Journal of Dental Research*, 2005,84(2):183-188.
49. María A L, Ricardo L M, Mariana P. Effect of photopolymerization time on the microhardness of resin cement beneath feldspathic ceramic. *Acta Odontol Latinoam*. 2023 Apr 29;36(1):40-46.
50. Barbon, F.J.; Isolan, C.P.; Soares, L.D.; Bona, A.D.; de Oliveira da Rosa,W.L.; Boscatto, N. A systematic review and meta-analysis on using preheated resin composites as luting agents for indirect restorations. *Clin. Oral Investig*. 2022, 26, 3383–3393
51. de Figueiredo EZ, de Souza Balbinot G, Castelo Branco Leitune V, et al. Niobium silicate as a filler for an experimental photopolymerizable luting agent. *J Prosthodont Res*. 2021 Feb 24;65(1):25-30.
52. Miitra, S. B. Chapter 2 – Nanoparticles for Dental Materials: Synthesis, Analysis, and Applications. *Emerging Nanotechnologies in Dentistry: Processes, Materials and Applications*.2012.15-33.

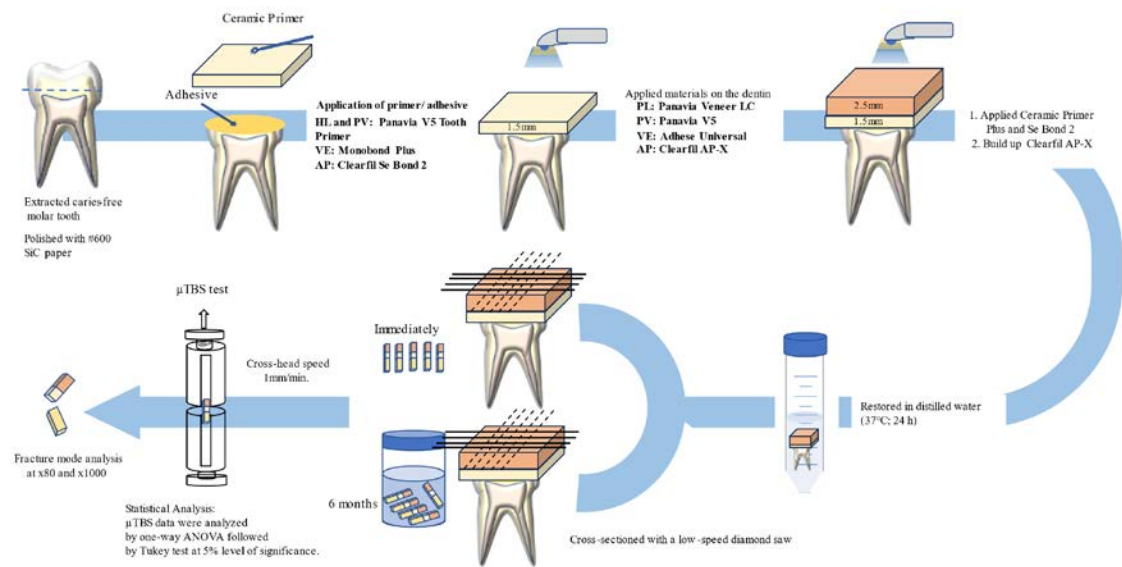


Figure 1. Graphical representation of specimen preparation and experimental set-ups for evaluating μ TBS and fracture mode.

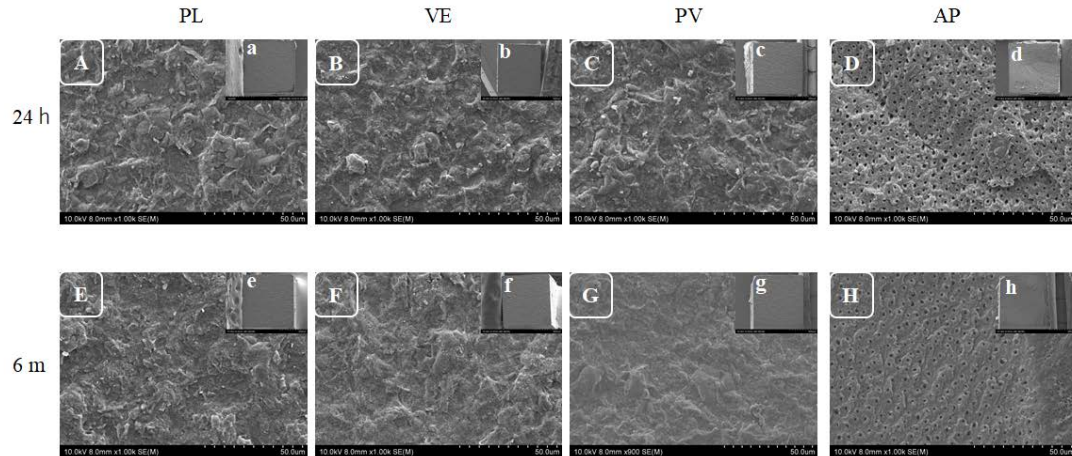


Figure 2. SEM images show the fractured surface following the μ TBS test ($\times 80$ and $\times 1000$). PL, VE, PV and AP mainly represent the cohesive failure after 24 hours and 6 months. (A, B, C, D, E, F, G, H) represents higher magnification of (a, b, c, d, e, f, g, h) respectively.

Table 1 Compositions of the materials tested.

Materials	Manufacturer	Compositions	Curing type
Panavia	Kuraray	silanated spherical silica, UDMA, Ytterbium trifluoride,	Light-cured

Veneer LC Lot# 5B0005	Noritake Dental, Tokyo, Japan	TEGDMA, hydrophilic aliphatic dimethacrylate, hydrophilic amide monomer, accelerators, dl-camphorquinone, pigments	
Variolink Esthetic LC Lot# Z03R50	Ivoclar Vivadent, Schaan, Liechtenstein	ytterbium trifluoride and spheroid mixed oxide, UDMA, Bis-GMA, HEMA, TEGDMA, and GDMA, initiator, stabilizer, pigments	Light-cured
Panavia V5 Lot# 8H0168	Kuraray Noritake Dental, Tokyo, Japan	Paste-A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica Paste-B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated aluminum oxide filler, accelerators, dl-camphorquinone, pigments	Dual-cured
Clearfil AP-X Lot# 850124	Kuraray Noritake Dental, Tokyo, Japan	Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, accelerators, pigments	Light-cured
Panavia V5 Tooth Primer Lot# AW0071	Kuraray Noritake Dental, Tokyo, Japan	MDP, HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water	
Adhese Universal Lot# Z027JP	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, HEMA, MDP, MCAP, decandiol dimethacrylate, dimethacrylate, ethanol, water, initiator, stabilizers, silicon dioxide	
Clearfil SE Bond 2 Lot# 4A0114, 4H0173	Kuraray Noritake Dental, Tokyo, Japan	Primer: MDP, HEMA, hydrophilic aliphatic dimethacrylate, dl-camphorquinone, hydrophobic aliphatic, water Bond: MDP, Bis-GMA, HEMA, dl-camphorquinone, hydrophobic aliphatic dimethacrylate, initiators, accelerators, silanated colloidal silica	
Clearfil Ceramic Primer Plus Lot# 2R0053	Kuraray Noritake Dental, Tokyo, Japan	Ceramic primer: 3-trimethoxysilylpropyl methacrylate, MDP, ethanol	
Monobond Plus Lot# Z038T1	Ivoclar Vivadent, Schaan, Liechtenstein	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate	

Bis-GMA: bisphenol-A-diglycidylmethacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloxydecyl dihydrogen phosphate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate.

Table 2 Mean $\pm$ SD (MPa) of microtensile bond strengths and fracture modes of resin materials.

Resin Cements	Mean $\pm$ SD (24 h) (n = 15)	Fracture Mode (24 h) A/C/D/Mixed/R	Mean $\pm$ SD (6 months) (n=15)	Fracture Mode (6 months) A/C/D/Mixed/R
PL	58.72 $\pm$ 9.53 ^{cd}	0/10/0/2/3	59.13 $\pm$ 7.01 ^{cd}	0/10/0/3/2
VE	17.62 $\pm$ 3.08 ^a	1/14/0/0/0	10.65 $\pm$ 3.72 ^a	2/13/0/0/0
PV	39.45 $\pm$ 6.27 ^b	0/13/0/2/0	53.26 $\pm$ 10.17 ^c	2/11/1/1/0
AP	63.14 $\pm$ 8.22 ^d	1/3/5/2/4	61.98 $\pm$ 10.12 ^d	0/4/7/0/4

The same letter indicates no statistical difference. The different letters indicate statistically significant differences ($p < 0.05$). A: fracture failure in adhesive; C: fracture failure in cement; D: fracture failure in dentin; Mixed: mixed failure in cement and resin block; R: fracture failure in resin composite.

Table 3 Means $\pm$ SD of KHN of the resin materials.

Resin Cements	KHN (24 h) (n = 15)	KHN (6 months) (n=15)
PL	28.45 $\pm$ 1.31 ^{bc}	30.41 $\pm$ 2.58 ^c
VE	31.03 $\pm$ 2.46 ^c	33.19 $\pm$ 1.35 ^c
PV	22.38 $\pm$ 2.23 ^a	24.81 $\pm$ 1.19 ^{ab}
AP	98.90 $\pm$ 4.76 ^d	100.70 $\pm$ 4.74 ^d

The same letter indicates no statistical difference. The different letters indicate statistically significant differences ($p < 0.05$).

Table 4 Mean $\pm$ SD (MPa) of cohesive strength of resin materials.

Resin Cements	Mean $\pm$ SD (24 h) (n = 15)	Mean $\pm$ SD (6 months) (n=15)
PL	55.64 $\pm$ 6.86 ^a	54.63 $\pm$ 11.97 ^a
VE	54.92 $\pm$ 5.54 ^a	58.56 $\pm$ 6.68 ^a
PV	76.21 $\pm$ 8.17 ^b	76.99 $\pm$ 4.23 ^b
AP	115.22 $\pm$ 12.76 ^c	113.90 $\pm$ 11.32 ^c

The same letter indicates no statistical difference. The different letters indicate statistically significant differences ($p < 0.05$).