Article category: Original article

Article title: Evaluation of Long-Term Bond Strength and Mechanical Properties of A Recently Launched Light-cure Resin Cement for Veneer Cementation

Author list:

Hong Zhang [1,2] ・ Yuan Yuan [3] ・ Yu Toida [1] ・ Papichaya Intajak [4] ・ Hidehiko Sano [1] ・Atsushi Tomokiyo [1]

Affiliations:

[1] Department of Restorative Dentistry, Faculty of Dental Medicine, Hokkaido University, Kita 13, Nishi 7, Sapporo 060-8586, Japan

[2] Department of Prosthodontics, Nanjing Stomatological Hospital, Affiliated Hospital of Medical School, Research Institute of Stomatology, Nanjing University, Nanjing 210008, China

[3] School/Hospital of Stomatology, Zhejiang Chinese Medical University, Hangzhou 310053, Zhejiang, China

[4] Department of Conservative Dentistry and Prosthodontics, Faculty of Dentistry, Srinakharinwirot University, 114 Sukhumvit 23, Wattana, Bangkok 10110, Thailand

Corresponding author:

Yuan Yuan

E-mail address: yyuann@outlook.jp

School/Hospital of Stomatology, Zhejiang Chinese Medical University, Hangzhou 310053, Zhejiang, China

Telephone number: +86 0571-8660-7899

Abstract

Objectives This study aimed to evaluate the initial and six-month bonding performance and mechanical properties of a recently launched light-cure resin cement compared to light-cure, dual-cure resin cements, and resin composite used for veneer cementation.

Materials and methods Four luting cements—Panavia Veneer LC (PVL), Variolink Esthetic LC (VEL), Panavia V5 (PV5), and Clearfil AP-X (APX)—were tested for microtensile bond strength (μTBS), Knoop hardness (KHN), degree of conversion (DC), and ultimate tensile strength (UTS) at 24-hour and 6-month. All values were subjected to statistical analysis ($\alpha = 0.05$). Fracture mode and resin-dentin interfaces were observed via SEM.

Results One-way ANOVA revealed significant differences among the materials for each measured property at 24-hour and 6-month ($p < 0.001$). The PVL group demonstrated superior bond strength at both time points (24-hour: 58.72 MPa; 6-month: 59.13 MPa), comparable to APX. In contrast, the VEL group showed a significant decrease in μTBS from 24-hour (27.62 MPa) to 6-month (10.65 MPa) ($p < 0.001$). The dual-cure cement PV5 showed marked improvements in μTBS and mechanical properties beyond 24 hours ($p < 0.001$). KHN and UTS were highest in APX. PVL and APX had higher DC values than VEL and PV5. SEM revealed variations in resin-dentin interfaces across the groups.

Conclusions PVL demonstrated reliable and durable bonding performance, while PV5 showed improvements after 24 hours, suggesting the need for extended curing time for optimal performance.

Clinical relevance PVL provides durable bonding and mechanical performance, making it a promising option for veneer cementation in clinical practice

Keywords: Luting cement・Bond strength・Knoop hardness・Degree of conversion・Nanocluster

Introduction

With CAD/CAM technology advancements and the development of resin-impregnate blocks, indirect porcelain, and resin laminate veneers have increased in popularity in clinical practice [1-3]. Additionally, veneer restorations offer several advantages, including minimal tooth preparation, sufficient strength, and cost-effectiveness, while their semi-translucent properties contribute to superior esthetic outcomes [4-6]. For optimal bonding and stress reduction on the restoration, veneer preparation should ideally remain confined to enamel. However, in practice, dentin exposure—particularly in cervical and proximal areas—is often unavoidable [2,7]. As a result, the cementation procedure becomes even more critical with exposed dentin surfaces [7-8]. The complexity arises from the dentin's lower inorganic content, variable tubular architecture, and outward intratubular fluid movement, which complicate the bonding process [9-10].

The selection of appropriate resin cement is critical to the success of veneers, with dual-cure and light-cure resin cements being two widely utilized options [11-14]. Dual-cure resin cements are popular due to their ability to cure through either light or self-curing, offering versatility for various types of veneers and adaptability in different clinical situations [15-16]. However, the limited working time and color instability of dual-cure resin cements present challenges in achieving optimal aesthetic outcomes and restoration longevity [17-18]. On the other hand, light-cure resin cements address these issues but rely solely on light for polymerization. This reliance can result in incomplete polymerization, particularly in deeper or more opaque areas, leading to compromised bond strength [12, 19-20]. Furthermore, the quality of polymerization is crucial for forming a durable resin-dentin bond [21]. Incomplete or uneven polymerization not only negatively affects bond strength but also accelerates the hydrolytic degradation of the bond over time, ultimately reducing the longevity of the restoration [22].

While numerous studies have focused on the bonding between veneers and enamel, research on the bonding between veneers and dentin remains relatively scarce [7,8]. Moreover, few studies have directly compared the performance of dual-cure and light-cure resin cements in bonding veneers to dentin [23], and even fewer have investigated the long-term durability of the bond for both cement types. Panavia Veneer LC (PVL) is a recently launched light-cure resin cement developed by Kuraray Noritake Dental. It features nanocluster technology to enhance polymerization and mechanical properties [24]. A recent in vitro study demonstrated that PVL exhibited better initial bonding performance than dual-cure resin cement [23]. However, its long-term durability remains underexplored and warrants further investigation.

Therefore, this study selected PVL for comparison with Variolink Esthetic LC (VEL), Panavia V5 (PV5), and Clearfil AP-X (APX) to represent different categories of luting agents: light-cure, dual-cure, and composite-based resin cements. The aim was to evaluate the six-month microtensile bond strength (μTBS) of three different resin cements and one resin composite bonded between dentin and a CAD/CAM resin slab, as well as to assess their Knoop hardness (KHN), degree of conversion (DC) and ultimate tensile strength (UTS). The null hypotheses of this research were: (i) there are no statistically significant differences in μTBS, KHN, UTS, and DC values among the four tested materials, and (ii) aging duration (24 hours vs. 6 months) does not significantly affect the μTBS, KHN, UTS, and DC values of these materials.

Materials and methods

The teeth were collected under review and approved by the Hokkaido University Research Ethics Committee (approval number 2018-9). Forty-eight extracted sound human third molars were stored in 0.5% Chloramine-T solution at 4°C and utilized within 6 months after extraction. Of these, twenty-four teeth were allocated for the μTBS test, eight for scanning electron microscopy (SEM) observation of resin-dentin interfaces, and sixteen for micro-Raman analysis. The sample size was determined based on a power analysis ($\alpha = 0.05$, power = 0.8) using previous studies on resin cement bond strength [23]. A minimum of 12 specimens per group was required to detect statistically significant differences.

Three resin cements and one resin composite were applied in this research as follows: (1) Panavia Veneer LC (PVL), (2) Variolink Esthetic LC (VEL), (3) Panavia V5 (PV5), and (4) Clearfil AP-X (APX). All the material compositions and polymerization systems are listed in Table 1.

Microtensile bond strength test

A schematic diagram of the experimental procedure is presented in Fig. 1. Twenty-four sound human third molars and Katana Avencia Block 2 (shade A3LT) were used. The CAD/CAM resin blocks were cut into 1.5-mm-thick slices. The surfaces of these slices were polished using 600-grit silicon carbide paper (SiC; Fuji Star, Sankyo-Rikagaku, Tokyo, Japan) under running water for 60 seconds. After polishing, the surfaces were treated with airborne-particle abrasion using 50 μm aluminum oxide powder at a pressure of 0.2 MPa with a laboratory blaster (Hiblaster Ovaljet, Shofu, Tokyo, Japan). The slices were then cleaned in an ultrasonic bath for 2 minutes and dried using an air syringe for 10 seconds. Each surface of the CAD/CAM composite resin slabs was treated with phosphoric acid for 5 seconds, rinsed with running water for 20 seconds, and subsequently salinized with a coupling agent according to the manufacturer's instructions: Clearfil Ceramic Primer Plus was applied for the PVL, PV5, and APX groups, while Monobond Plus was used for the VEL group.

All the teeth were randomly assigned to four groups according to the tested luting cements: PVL, VEL, PV5, and APX. The middle occlusal dentin surface was obtained by removing the coronal enamel with a gypsum model trimmer under running water coolant. The exposed dentin surfaces were then polished with 600-grit SiC paper for 60 seconds under running water to create a standardized smear layer. For dentin pretreatment, Panavia V5 Tooth Primer was applied in the PVL and PV5 groups, Adhese Universal in the VEL group, and Clearfil SE Bond 2 (SE2) in the APX group, all according to the respective manufacturer's instructions.

Following the priming and bonding procedures, the four luting cements were applied to the dentin surfaces, and the CAD/CAM resin slices (1.5-mm-thick) were firmly positioned over the cement. The assemblies were then light-cured for 40 seconds. Subsequently, to standardize the specimen thickness and ensure uniform stress distribution during microtensile bond strength testing, Clearfil AP-X resin composite layers were built up on the resin slices to achieve a final height of 4 mm for the μTBS test. Standardizing sample geometry minimizes variability in testing conditions, making the results more reliable and comparable. A light curing unit (Pencure 2000, Morita, Tokyo, Japan) was used with an output irradiance of 2000 mW/cm2. All specimens were stored in distilled water at 37°C for 24 hours.

Each specimen was sectioned perpendicular to the bonded surface with a low-speed diamond saw to obtain resin-dentin beams (1±0.05 mm^2). The beams from three specimens in each group were stored in 37℃ distilled water for 6 months and the beams from the other three specimens were tested for μTBS immediately. Five beams at the central area from each specimen were selected for the μTBS test (n = 15). The beams were fixed to a Ciucchi's jig with cyanoacrylate glue (Model Repair 2 Blue, Dentsply-Sankin, Tokyo, Japan). A standard μTBS test was performed in approximately 1 mm^2 nontrimmed beams verified by a digital caliper and subjected to a tensile force at a crosshead speed of 1 mm/min crosshead speed by using a desktop testing apparatus (EZ-S, Shimadzu, Kyoto, Japan) until fracture occurred [25-26]. As a result, fifteen μTBS values for each tested group were evaluated.

Fracture mode analysis

The fracture modes were first observed by a light microscope (×20 magnification; Magnifier Light, Asone, Osaka, Japan). Then, the specimens were air-dried, mounted on an aluminum stub, and sputter-coated with Pt-Pd for 120 seconds, and morphological differences were examined by using 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: adhesive failure; cohesive failure in cement; cohesive failure in dentin; mixed failure in cement and resin block; and cohesive failure in resin composite [26]. A single operator performed the fracture mode analysis blindly.

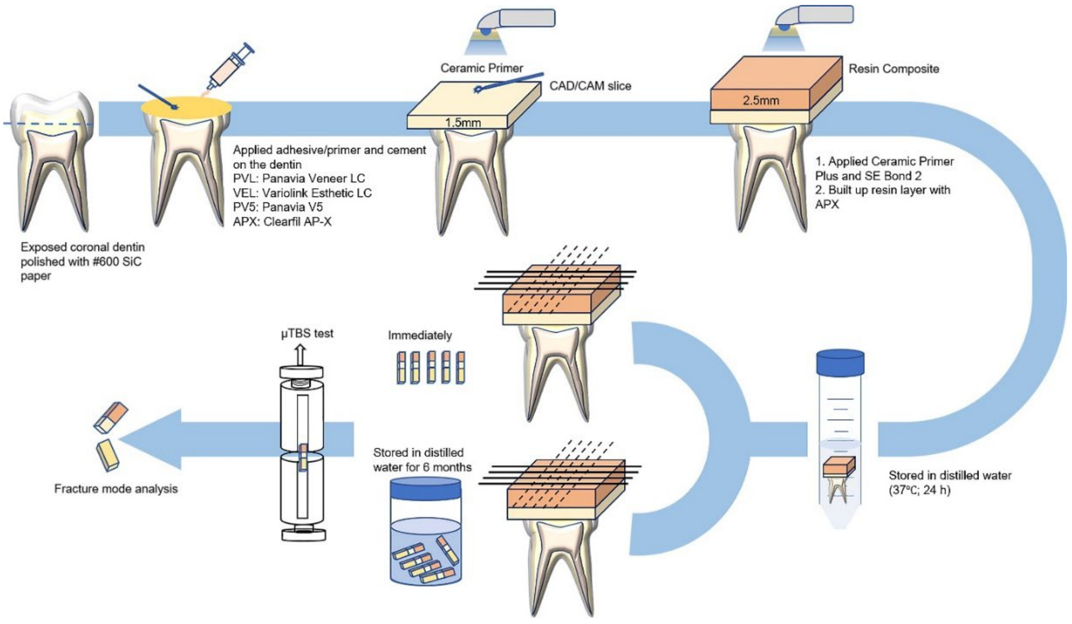

Figure. 1 Schematic diagram of the study design for evaluating μTBS and fracture mode.

Table 1 Chemical composition and application mode of the materials used in this study

Materials	Manufacturer	Compositions	Abbreviation
Panavia Veneer LC Lot# 5B0005	Kuraray Noritake Dental, Tokyo, Japan	Silanated spherical silica, UDMA, ytterbium trifluoride, TEGDMA, hydrophilic aliphatic dimethacrylate, hydrophilic amide monomer, accelerators, dl-camphorquinone, pigments	PVL
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	VEL
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 fluoroalminosilicate 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	PV5

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	APX
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	SE2
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	
Katana Avencia Block 2 Lot# 001122	Kuraray Noritake, Tokyo, Japan	Mixed filler with colloidal silica and aluminum oxide, cured resins consisting of methacrylate monomer (copolymer of UDMA and other methacrylate monomers), pigments	

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

Observation of resin-dentin interface

Eight teeth were bonded with four resin materials in the same manner as the µTBS test, with two teeth per group: one tooth for the 24-hour assessment and one for the 6-month assessment. All teeth were stored in distilled water at 37°C for 24 hours before sectioning. Each tooth was sectioned perpendicular to the resin-dentin interface to obtain 2-mm-thick slabs, with two slabs being obtained from each tooth. The specimens for the 6-month assessment were stored in distilled water at 37°C until SEM preparation. All the slabs were prepared following a protocol outlined by Yuan et al [27]. After that, the specimens were sputter coated with Pt-Pd and observed by SEM with an accelerating voltage of 10 kV.

Knoop hardness measurement

A transparent silicon mold was fabricated with a 2-mm-height and 10-mm-diameter cavity. The mold was filled with resin materials, and confined between two polyester strips and glass microscope slides to extrude excess and prevent an oxygen inhibition layer. Then light cured for 20 seconds on each top and bottom side of samples with a cordless LED at 2000 mW/cm^2. During the light irradiation, the light area was positioned in the center of the specimen. A filter was employed to hold ambient red light. Twelve specimens were prepared for each group. Half of the samples were tested after 24 hours meanwhile the others were tested after 6 months of storage in a dry, dark environment at 37°C.

Indentations were sequentially performed using a microhardness tester (MVK-C, Akashi, Kanagawa, Japan) with a 20× objective lens and a 50 g load for 15 seconds. Ten indentations were made at top surfaces at 0.5 mm intervals for every sample, beginning from the middle and proceeding outward. The long-axis length of each indentation was measured and recorded. An average of the ten measurements was calculated on each surface and transformed into KHN using the following formula:

$$KHN = 14230 \times \frac{F}{d^2}$$

F is the force in grams (50 g) and d is the length of the longest diagonal in micrometers.

Micro-Raman analysis

Sixteen teeth were randomly assigned into four groups: PVL, VEL, PV5, and APX. The teeth were prepared and bonded as previously mentioned for the μTBS test. Each group was further divided into two subgroups based on storage time in distilled water at 37°C: 24 hours and 6 months. Each specimen was then sectioned perpendicularly to the bonding interface using a low-speed diamond blade to obtain two slabs. The slabs were polished with 800, 1000, 1200, and 1500-grit SiC paper followed by polishing with 6, 3, and 1μm diamond paste (DP-Paste, Struer, Copenhagen, Denmark). After polishing, the slabs were ultrasonically cleaned for 3 minutes.

The slabs were analyzed using a computer-controlled confocal laser Raman spectro-microscope (Horiba Scientific XploRA, Villeneuved'Ascq, France). The micro Raman spectrometer was first calibrated relative to the main Si peak (521 cm^{-1}). Samples were analyzed using the following parameters: a laser wavelength of 532 nm, a grating of 2400 gr/mm, a range of 400 to 2000 cm^{-1}, a Czerny-Turner type spectrograph with F = 200 mm, a ×100 magnification objective lens, an exposure time of 10 seconds, and a scan accumulation count of 8. Five spots were detected in each slab, and twenty DC values for each group were evaluated.

The values of DC were calculated according to the following formula:

$$DC\% = \left(1 - \frac{R(cured)}{R(uncured)}\right) \times 100$$

R is the ratio of the peak intensities of aliphatic to aromatic groups, measured around 1639 cm^{-1} and 1609 cm^{-1}. This ratio is assessed in uncured states and after curing for 24 hours or 6 months.

Ultimate tensile strength test

A transparent silicon mold with a length of 20 mm and a width of 2 mm was customized for the study. The four resin materials under test were dispensed into the mold and light-cured for 20 seconds. The cured specimens were trimmed to a dumbbell shape with a neck diameter of 1 mm and stored in distilled water at 37°C. Twelve samples were prepared for each group. After either 24 hours or 6 months of distilled water storage at 37°C, the specimens were subjected to a μTBS test to assess UTS, following the previously described μTBS testing procedures.

Statistical Analysis

Data for the measured properties were statistically analyzed using the SPSS software package (SPSS version 25; IBM Corp, Armonk, NY, USA). Shapiro-Wilk test values for normal distribution and Levene's test values for homogeneity of variance of measurements were evaluated. Based on these results, μTBS, KHN, DC, and UTS values were analyzed using one-way ANOVA followed by Tukey's HSD post-hoc test, conducted separately for the 24 hours and 6 months groups (α=0.05). To evaluate the differences in the properties of the same material between two independent time points (24 hours and 6 months), an independent Student's t-test was employed(α=0.05).

Results

Microtensile bond strength test

The μTBS values are presented in Table 2. One-way ANOVA revealed a significant effect of material type on μTBS values at 24 hours (p<0.001) and 6 months (p<0.001). The t-test comparison of μTBS values between the 24 hours and 6 months indicated significant differences for the VEL and PV5 groups (p<0.001), while no significant differences were observed for the PVL (p=0.894) and APX (p=0.734) groups.

Table 2 Mean±SD (MPa) of μTBS of four luting cements at 24-hour and 6-month

Luting Cement	μTBS	
	24 h	**6 m**
PVL	58.72 ± 9.53^{a}	59.13 ± 7.01^{AB}
VEL	27.62 ± 3.08^{c}*	10.65 ± 3.72^{C}*
PV5	39.45 ± 6.27^{b}*	53.26 ± 10.17^{B}*
APX	63.14 ± 8.22^{a}	61.98 ± 10.12^{A}

Different lowercase superscripts denote statistically significant differences among materials at 24-hour and different uppercase superscripts indicate significant differences among materials at 6-month (p<0.001). Asterisks (*) indicate significant differences between 24-hour and 6-month values for the same material (p<0.001).

Fracture mode analysis

The failure modes are presented in Fig. 2, with no pretest failures observed. SEM images of the representative fracture modes on the dentin side (x80 and x1000 magnifications) were displayed in Fig. 3. At 24-hour, cement failure was the predominant fracture mode in the PVL (66.67%), VEL (93.33%), and PV5 (86.67%) groups. Similar results were observed at the 6-month, with cement failure occurring in PVL (66.67%), VEL (86.67%), and PV5 (73.33%). Notably, the PVL group exhibited no adhesive failure or dentin failure at either 24-hour or 6-month. The PV5 group had no resin composite failure at either 24-hour or 6-month. The APX group demonstrated a higher incidence of dentin failure than other groups, with 33.33% at 24-hour and 46.47% at 6-month.

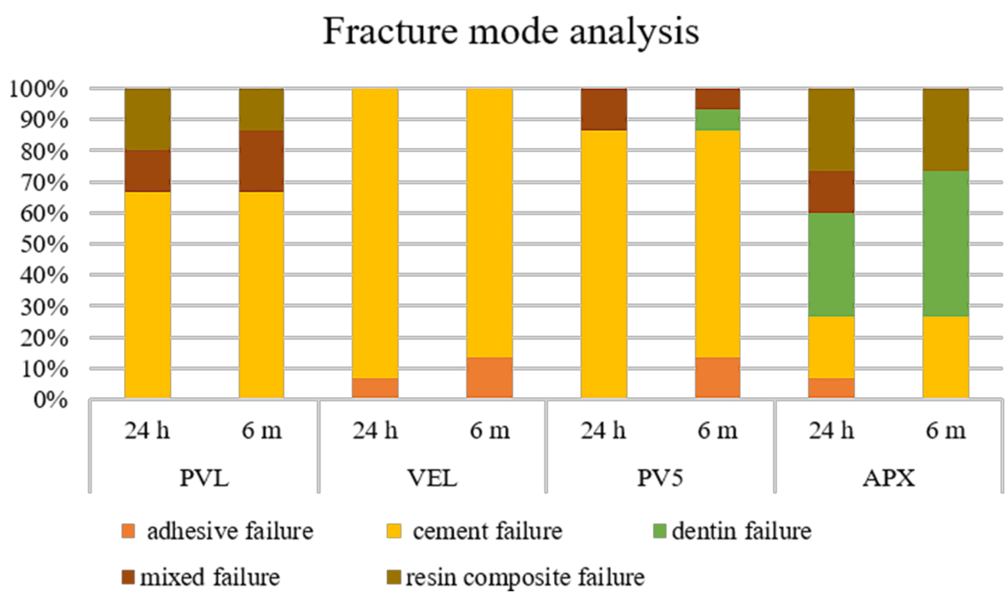

Figure. 2 Failure mode (%) of four luting materials at 24-hour and 6-month.

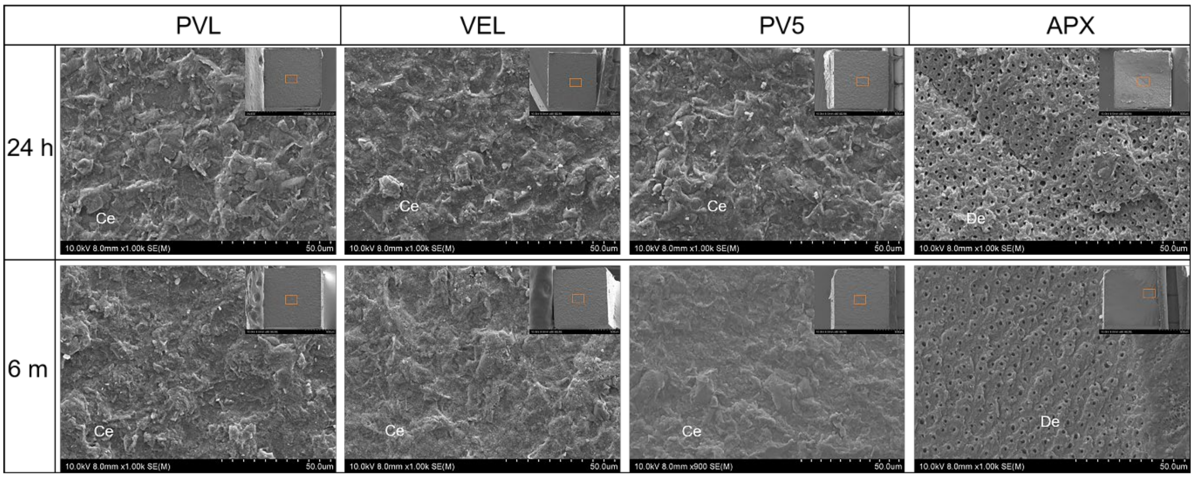

Figure. 3 Represent SEM images show the fractured surface following the μTBS test (×80 and ×1000) at 24-hour and 6-month. The PVL, VEL, and PV5 groups typically exhibited fracture failure in the cement, whereas the APX group primarily showed fracture failure in the dentin at 24-hour and 6-month. Ce: Cement; De: Dentin.

Observation of resin-dentin interface

Typical resin-dentin interface images are shown in Figure. 4. The average cement layer thickness was highest in the APX group, followed by the PVL group, with thinner layers observed in the VEL and PV5 groups. Resin tags extending into the dentin were visible in all groups; however, the VEL group exhibited fewer and shorter tags compared to the PVL, PV5, and APX groups. After 6 months, gaps between the resin cement and CAD/CAM resin block were noted in both the PVL and PV5 groups, while a gap between the dentin and resin layer was observed in the VEL group.

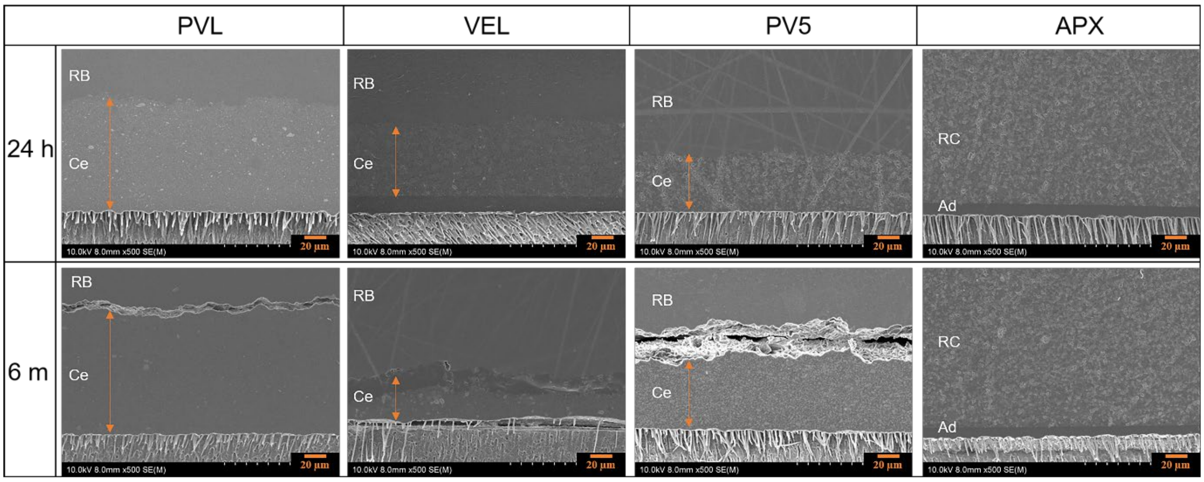

Figure. 4 SEM images displaying the resin-dentin interface for each group (×500) at 24-hour and 6-month. In the PVL group, a uniform cement layer was observed at 24-hour and 6-month, while the VEL group showed variations

in cement layer thickness. Gaps between the resin cement and CAD/CAM resin block were observed in the PVL and PV5 groups, whereas a gap between the dentin and resin layer was observed in the VEL group. In the APX group, the resin cement layer was significantly thicker, measuring over 100 µm. RB: CAD/CAM Resin Block; Ce: resin cement; RC: Clearfil AP-X Resin Composite; Ad: Adhesive.

Knoop hardness measurement

The KHN results for each material at 24 hours and 6 months are presented in Table 3. One-way ANOVA showed significant differences in KHN among materials at both 24 hours ($p<0.001$) and 6 months ($p<0.001$). The t-test comparison of KHN values between 24 hours and 6 months for each material indicated no significant changes for PVL ($p=0.127$), VEL ($p=0.089$), and APX ($p=0.527$). However, PV5 showed a significant increase in hardness over time ($p<0.001$).

Micro-Raman analysis

The DC values are presented in Table 3. One-way ANOVA revealed significant differences among the materials at both 24 hours ($p<0.001$) and 6 months ($p=0.007$). The t-test comparison of DC values between 24 hours and 6 months indicated a significant increase for PV5 ($p<0.001$), with no significant changes for PVL ($p=0.226$), VEL ($p=0.231$), and APX ($p=0.668$).

Table 3. Mean±SD of KHN and DC of four luting cements at 24-hour and 6-month

Luting Cement	KHN (HK)		DC (%)	
	24 h	6 m	24 h	6 m
PVL	28.45 ± 1.31^{b}	30.41 ± 2.58^{A}	61.45 ± 6.31^{a}	60.41 ± 5.58^{AB}
VEL	31.03 ± 2.46^{b}	33.19 ± 1.35^{A}	50.03 ± 5.46^{b}	48.19 ± 4.35^{A}
PV5	$22.38 \pm 2.23^{a*}$	$34.81 \pm 1.19^{A*}$	$45.38 \pm 6.23^{b*}$	$57.81 \pm 6.19^{B*}$
APX	98.90 ± 4.76^{c}	100.70 ± 4.74^{B}	58.90 ± 5.76^{a}	59.70 ± 4.74^{B}

Different lowercase superscripts denote statistically significant differences among materials at 24-hour and different uppercase superscripts indicate significant differences among materials at 6-month ($p<0.001$). Asterisks (*) indicate significant differences between 24-hour and 6-month values for the same material ($p<0.001$).

Ultimate tensile strength test

The UTS results are presented in Table 4. One-way ANOVA showed significant differences among the materials at both 24 hours ($p<0.001$) and 6 months ($p<0.001$). The t-test comparison of UTS values between 24 hours and

6 months revealed a significant increase for PV5 ($p<0.001$), while no significant changes were observed for PVL ($p=0.861$), VEL ($p=0.328$), and APX ($p=0.853$).

Table 4. Mean±SD (MPa) of UTS of four luting cements at 24-hour and 6-month.

Luting Cement	UTS	
	24 h	**6 m**
PVL	55.64 ± 6.86^{a}	54.63 ± 11.97^{A}
VEL	54.92 ± 5.54^{a}	58.56 ± 6.68^{A}
PV5	$56.21 \pm 8.17^{a*}$	$76.99 \pm 4.23^{B*}$
APX	115.22 ± 12.76^{b}	113.90 ± 11.32^{C}

Different lowercase superscripts denote statistically significant differences among materials at 24-hour and different uppercase superscripts indicate significant differences among materials at 6-month ($p<0.001$). Asterisks (*) indicate significant differences between 24-hour and 6-month values for the same material ($p<0.001$).

Discussion

Light-cure and dual-cure resin cements are commonly employed for luting veneers [28]. The choice of cement depends on the veneer's characteristics, such as opacity and thickness, as well as the bonding requirements of the tooth surface. Color stability and bonding performance are crucial considerations for selecting an appropriate cement [29]. This study assessed the initial and six-month bonding performance and mechanical properties of resin cements with different curing systems. The results of this study demonstrated statistically significant effects of material type on µTBS, KHN, DC, and UTS values, as confirmed by one-way ANOVA at both 24-hour and 6-month ($p<0.001$), leading to the rejection of the first null hypothesis. Additionally, t-tests indicated that, while the PVL, VEL, and APX groups exhibited no significant changes in µTBS, KHN, DC, and UTS values between 24-hour and 6-month ($p>0.05$), the PV5 group—a dual-cure resin cement—demonstrated a significant increase in µTBS, KHN, and DC over time ($p<0.001$). Moreover, the VEL group revealed a significant decrease in µTBS ($p<0.001$). Hence, the second null hypothesis was partially rejected.

The µTBS of the dual-cure resin cement PV5 was initially significantly lower than PVL and APX but showed a notable increase after 6 months (Table 2). This result highlights the durability and adaptability of PV5, suggesting that extended post-curing reactions may enhance its mechanical and bonding properties over time. The improvement in bond strength aligns with the observed increase in DC (Table 3) and is consistent with findings from previous studies [23,30]. Furthermore, prior research has confirmed the durable bonding performance of PV5, which supports the findings of this study [31-34]. Therefore, careful consideration of initial retention rates is essential when using dual-cure resin cements in clinical applications, as initial bond strength may be lower despite later improvements.

Clearfil AP-X resin composite was chosen for this study due to its established use as a luting cement in both in vitro and in vivo studies [35-38]. It is commonly used with SE2 in μTBS tests and has demonstrated satisfactory and durable bond strength [36, 39-40], which aligns with the results of the present study. It is probably due to SE2 enhancing the quality of the hybrid layer and improving the acid-base resistant zone at the resin-dentin interface [41-42]. The light-initiated system may also improve μTBS and UTS (Table 2, 4). UTS, reflecting the material's maximum load-bearing capacity under tensile stress, is closely linked to both adhesive cohesion and the overall mechanical stability of the material [43]. Improving UTS is commonly achieved by increasing filler volume, adjusting particle sizes, and modifying filler shapes to enhance mechanical performance [44-45]. A correlation between filler volume and UTS was observed in this study. Specifically, APX exhibited the highest UTS, followed by PV5 (38 vol%, 61 wt%), which is consistent with their respective filler contents. Additionally, APX's high filler content of 70 vol% (86 wt%) and its average particle size of 3 μm contributed to the increased KHN (Table 3). This result suggests that an increased quantity of particle fillers contributes to improved mechanical properties, as the incorporation of fillers enhances the internal cohesion and strength of the material. Although hardness is reported to be closely related to the DC [30], APX showed no significant difference in DC but significantly higher KHN compared to the other three luting cements in this study (Table 3). This may be attributed to APX exceeding the recommended luting thickness due to its high viscosity, as shown by the thickness exceeding 100 μm in Figure 4, which could affect polymerization [29].

Comparable to APX, the light-cure resin cement PVL demonstrated satisfactory and durable μTBS and DC (Table 2, 3), probably due to its effective light-activated polymerization and innovative nanocluster technology [23,46]. The new spherical silica fillers in PVL may enhance the light transmission and polymerization kinetics, improving bond strength and durability [47-48]. PVL utilized nano- and micro-sized spherical fillers, which may have contributed to its improved UTS values. PVL demonstrated a higher filler content (47 vol% / 66 wt%) than VEL (38 wt%) [49] but showed similar UTS values, which may be due to the presence of nano- and micro-sized fillers. Additionally, the nanocluster flowable fillers and the high filler content help achieve a consistent and uniform resin layer, as evidenced by the SEM images of the dentin-resin interface (Fig. 4).

In contrast, another light-cure resin cement VEL exhibited similar KHN and UTS, but its μTBS were significantly lower than those of PVL. In our study, direct light was applied to the specimens for KHN and UTS testing, whereas for μTBS, the luting cements were positioned beneath the CAD/CAM block, reducing direct light exposure. Therefore, it is probable that the VEL initiator is more sensitive to light-curing attenuation compared to PVL. Additionally, a relatively higher percentage of adhesive failure was observed (Fig. 2, 3) and the weakest part of the resin-dentin interface was located between the dentin and adhesive layer in the VEL group (Fig. 4). This may imply that the universal adhesive used in the VEL group lacks sufficient long-term durability. These findings align with previous studies that reported a decline in bond strength after 1 year of water storage or thermocycling [50-51]. Furthermore, an insufficient DC could increase water permeation, accelerating the degradation of the luting cement, as supported by previous studies on water absorption's impact on adhesive and hybrid layer degradation [52-53]. However, Carek et al. reported that the DC of VEL increased after 24 hours, which differs from the present study finding [54]. This discrepancy may be due to variations in vibrational spectroscopy techniques, specimen preparation, and material compositions across studies [55].

This in vitro study evaluated the performance of dual-cure and light-cure resin cements, along with a resin composite, for veneer cementation. The PVL demonstrated superior bond strength comparable to APX at 24 hours and 6 months. This finding may have important clinical implications, particularly in cases with extensive dentin exposure and high bond strength requirements. In such situations, PVL could be a viable option due to its strong initial polymerization, eliminating the need for preheating. This feature ensures that PVL can achieve optimal bonding strength early in the procedure, offering efficiency, reliability, and clinical ease of use. In contrast, while PV5 exhibited lower bond strength than PVL and APX at 24 hours, it showed a notable improvement after 6 months, with bond strength comparable to PVL. This time-dependent increase in bond strength suggests that PV5 could be particularly effective in clinical cases where long-term performance is prioritized, especially in areas where light-curing may be less efficient. Although resin cements are generally considered to reach optimal bond strength within 24 hours, the present study indicates that PV5 may require more than 24 hours to fully achieve stable bond strength. Clinically, this suggests that for restorations cemented with PV5, it may be beneficial to allow additional curing time before subjecting the restoration to heavy masticatory forces. Such a precaution could help ensure the long-term durability and performance of the bonded restoration.

However, the six-month aging period may not fully reflect the materials' true long-term performance. Future studies with extended observation periods are necessary to validate these findings over time. Additionally, this in vitro setup cannot account for the variability and complexity of the oral environment, including factors like salivary enzymes, thermal cycling, and mechanical stresses, which may influence bond durability in clinical settings.

Further research is needed to assess these effects and explore the impact of clinical variables on the longevity and reliability of the materials evaluated here.

Conclusions

Within the limitations of this in vitro study, the light-cure resin cement PVL demonstrated reliable and durable bonding performance and mechanical properties over time. Its superior bond strength and mechanical properties make it a promising option for clinical veneer cementation, particularly in situations requiring consistent, high-performance bonding. However, further studies with extended observation periods are necessary to assess its long-term clinical performance. Notably, the dual-cure resin cement PV5 showed improvements in bond strength and mechanical properties beyond 24 hours, suggesting that extended curing time may enhance its performance, especially in deeper or more challenging restoration areas. Future research should focus on exploring these findings in clinical settings to evaluate the impact of extended curing times on working time optimization and bond strength in complex procedures.

Funding This research was supported by a grant in aid for scientific research from the Ministry of Education, Science, Sports and Culture of Japan (#22K09994); '3456' Cultivation Program For Junior Talents of Nanjing Stomatological School, Affiliated Hospital of Medical School, Nanjing University [0222C106] and the Jiangsu Seniors Health Research Foundation [LKM2022039].

Author Contributions H.Z. and Y.Y. performed the experiment, statistical analysis and wrote the manuscript. P.I. created the result tables and figures. T.Y performed the experiment. H.S. idea, experimental design and review the manuscript. A.T. review the manuscript. All authors reviewed the manuscript.

Data availability No datasets were generated or analyzed during the current study.

Declarations

Ethical Approval All procedures performed in this study were conducted under the principles outlined in the Declaration of Helsinki. Ethical approval for the study was granted by the Hokkaido University Research Ethics Committee (Approval Number: 2018-9).

Informed consent Not applicable.

Competing interests The authors declare no conflicts of interest.

References

1. Mainjot AK, Dupont NM, Oudkerk JC, Dewael T Y, Sadoun MJ (2016) From artisanal to CAD-CAM blocks: state of the art of indirect composites. J Dent Res 95: 487-495. https://doi.org/10.1177/0022034516634286

2. Araujo E, Perdigão J (2021) Anterior Veneer Restorations-An Evidence-based Minimal-Intervention Perspective. J Adhes Dent 23. https://doi.org/ 10.3290/j.jad.b1079529.

3. Awada A, Nathanson D (2015) Mechanical properties of resin-ceramic CAD/CAM restorative materials. J Prosthet Dent 114: 587-593. https://doi.org/10.1016/j.prosdent.2015.04.016

4. Alothman Y, Bamasoud MS (2018) The success of dental veneers according to preparation design and material type. Open Access Maced J Med Sci 6: 2402. https://doi.org/ 10.3889/oamjms.2018.353

5. Ravinthar K (2018) Recent advancements in laminates and veneers in dentistry. RJPT 11: 785-787. https://doi.org/ 10.5958/0974-360X.2018.00148.8

6. Fahl Jr N, Ritter AV (2021) Composite veneers: The direct–indirect technique revisited. J Esthet Restor Dent 33: 7-19. https://doi.org/10.1111/jerd.12696

7. Öztürk E, Bolay Ş, Hickel R, Ilie N (2013) Shear bond strength of porcelain laminate veneers to enamel, dentine and enamel–dentine complex bonded with different adhesive luting systems. J Dent 41: 97-105. https://doi.org/10.

1016/j.jdent.2012.04.005

8. Ibarra G, Johnson GH, eurtsen W, Vargas MA (2007) Microleakage of porcelain veneer restorations bonded to enamel and dentin with a new self-adhesive resin-based dental cement. Dent Mater 23: 218-225. https://doi.org/10.1016/j.dental.2006.01.013

9. De Munck JD, Van Landuyt K, Peumans M et al (2005) A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res 2005; 84: 118-132. https://doi.org/10.1177/154405910508400204

10. Perdigão J (2020) Current perspectives on dental adhesion:(1) Dentin adhesion–not there yet. Jpn Dent Sci Rev 56: 190-207. https://doi.org/10.1016/j.jdsr.2020.08.004

11. de Bragança GF, Mazão JD, Versluis A, Soares CJ (2021) Effect of luting materials, presence of tooth preparation, and functional loading on stress distribution on ceramic laminate veneers: A finite element analysis.

J Prosthet Dent 125: 778-787. https://doi.org/10.1016/j.prosdent.2020.02.005

12. Almeida JR, Schmitt GU, Kaizer MR, Boscato N, Moraes RR (2015) Resin-based luting agents and color stability of bonded ceramic veneers. J Prosthet Dent 114: 272-277. https://doi.org/10.1016/j.prosdent.2015.01.008

13. Stamatacos C, Simon JF (2013) Cementation of indirect restorations: an overview of resin cements. Compend Contin Educ Dent 34: 42-46.

14. Yu H, Zheng M, Chen R, Cheng H (2014) Proper selection of contemporary dental cements. Oral Health Dent Manag 13: 54-59.

15. El-Mowafy O (2001) The use of resin cements in restorative dentistry to overcome retention problems. J Can Dent Assoc 67: 97-102.

16. Attar N, Tam LE, McComb D (2003) Mechanical and physical properties of contemporary dental luting agents. J Prosthet Dent 89: 127-134. https://doi.org/10.1067/mpr.2003.20

17. Hardan L, Bourgi R, Hernández Escamilla T et al (2024) Color stability of dual-cured and light-cured resin cements: A systematic review and meta-analysis of in vitro studies. J Prosthodont 33: 212-220. https://doi.org/10.1111/jopr.13757

18. Marchionatti AME, Wandscher VF, May MM, Bottino MA, May LG (2017) Color stability of ceramic laminate veneers cemented with light-polymerizing and dual-polymerizing luting agent: A split-mouth randomized clinical trial. J Prosthet Dent 118(5): 604-610. https://doi.org/10.1016/j.prosdent.2016.11.013

19. Sinhoreti MAC, Manetta IP, Tango RN, Iriyama NT, Consani RLX, Correr-Sobrinho L (2007) Effect of light-curing methods on resin cement Knoop hardness at different depths. Braz Dent J 18: 305-308. https://doi.org/10.1590/S0103-64402007000400006

20. Ilday NO, Bayindir YZ, Bayindir F, Gurpinar A (2013) The effect of light curing units, curing time, and veneering materials on resin cement microhardness. J Dent Sci 8: 141-146. https://doi.org/10.1016/j.jds.2012.09.014

21. Cadenaro M, Maravic T, Comba A et al (2019) The role of polymerization in adhesive dentistry. Dent Mater 35(1): e1-e22. https://doi.org/10.1016/j.dental.2018.11.012

22. Foxton RM, Pereira PN, Nakajima M, Tagami J, Miura H (2002) Durability of the dual-cure resin

cement/ceramic bond with different curing strategies. J Adhes Dent 4(1): 49-45.

23. Kawamura M, Toida Y, Hoshika S et al (2022) Influence of Novel Experimental Light-Cured Resin Cement on Microtensile Bond Strength. Polymers (Basel) 14: 4075. https://doi.org/10.3390/polym14194075

24. https://panaviacements.com/panavia-veneer-lc/

25. Sano H, Chowdhury AFMA, Saikaew P, Matsumoto M, Hoshika S, Yamauti M (2020) The microtensile bond strength test: Its historical background and application to bond testing. Jpn Dent Sci Rev 56: 24-31. https://doi.org/10.1016/j.jdsr.2019.10.001

26. Armstrong S, Breschi L, Özcan M, Pfefferkorn F, Ferrari M, Van Meerbeek B (2017) Academy of Dental Materials guidance on in vitro testing of dental composite bonding effectiveness to dentin/enamel using micro-tensile bond strength (μTBS) approach. Dent Mater 33: 133-143. https://doi.org/10.1016/j.dental.2016.11.015

27. Yuan Y, Intajak P, Islam R et al (2023) Effect of sodium hypochlorite on bonding performance of universal adhesives to pulp chamber dentin. J Dent Sci 18: 1116-1124. https://doi.org/10.1016/j.jds.2022.11.007

28. Assaf A, Azer SS, Sfeir A, Al-Haj Husain N, Özcan M (2023) Risk Factors with Porcelain Laminate Veneers Experienced during Cementation: A Review. Materials 16: 4932. https://doi.org/10.3390/ma16144932

29. Sari T, Ural C, Yüzbasioglu E, Duran I, Cengiz S, Kavut I (2018) Color match of a feldspathic ceramic CAD-CAM material for ultrathin laminate veneers as a function of substrate shade, restoration color, and thickness. J Prosthet Dent 119: 455-460. https://doi.org/10.1016/j.prosdent.2017.02.022

30. De Souza G, Braga RR, Cesar PF, Lopes GC (2015) Correlation between clinical performance and degree of conversion of resin cements: a literature review. J Appl Oral Sci 23: 358-368. https://doi.org/10.1590/1678-775720140524

31. Maaßen M, Wille S, Kern M (2021) Bond strength of adhesive luting systems to human dentin and their durability. J Prosthet Dent 125: 182-188. https://doi.org/10.1016/j.prosdent.2019.07.012

32. Vergano EA, Baldi A, Comba A et al (2021) Bond Strength Stability of Different Dual-Curing Adhesive Cements towards CAD-CAM Resin Nanoceramic: An In Vitro Study. Appl Sci 11: 3971. https://doi.org/10.3990/app11093971

33. Takagaki T, Ko AK, Halabi S et al (2019) Adhesion durability of dual-cure resin cements and acid-base

resistant zone formation on human dentin. Dent Mater 35: 945-952. https://doi.org/10.1016/j.dental.2019.02.020

34. Malysa A, Wezgowiec J, Grzebieluch W, Danel DP, Wieckiewicz M (2022) Effect of Thermocycling on the Bond Strength of Self-Adhesive Resin Cements Used for Luting CAD/CAM Ceramics to Human Dentin. Int J Mol Sci 23: 745. https://doi.org/10.3390/ijms23020745

35. Politano G, Van Meerbeek B, Peumans M (2018) Nonretentive bonded ceramic partial crowns: concept and simplified protocol for long-lasting dental restorations. J Adhes Dent 20: 495-510. https://doi.org/10.3290/j.jad.a41630

36. Putzeys E, Vercruyssen C, Duca RC et al (2020) Monomer release from direct and indirect adhesive restorations: a comparative in vitro study. Dent Mater 36: 1275-1281. https://doi.org/10.1016/j.dental.2020.06.001

37. Falacho RI, Marques JA, Palma PJ et al (2022) Luting indirect restorations with resin cements versus composite resins: Effects of preheating and ultrasound energy on film thickness. J Esthet Restor Dent 34(4): 641-649. https://doi.org/10.1111/jerd.12851

38. Bortolotto T, Guillarme D, Gutemberg D, Veuthey JL, Krejci I (2013) Composite resin vs resin cement for luting of indirect restorations: comparison of solubility and shrinkage behavior. Dent Mater J 32(5): 834-838. https://doi.org/10.4012/dmj.2013-153

39. Ahmed MH, Yoshihara K, Mercelis B, Van Landuyt K, Peumans M, Van Meerbeek B (2020) Quick bonding using a universal adhesive. Clin Oral Investig 24: 2837-2851. https://doi.org/10.1007/s00784-019-03149-8

40. Kitahara S, Shimizu S, Takagaki T et al (2024) Dentin Bonding Durability of Four Different Recently Introduced Self-Etch Adhesives. Materials17:4296. https://doi.org/10.3390/ma17174296

41. Van Meerbeek B, Yoshihara K, Yoshida Y et al (2011) State of the art of self-etch adhesives. Dent Mater 27: 17-28. https://doi.org/10.1016/j.dental.2010.10.023

42. Yoshihara K, Nagaoka N, Nakamura A, Hara T, Yoshida Y, Van Meerbeek B (2021) Nano-layering adds strength to the adhesive interface. J Dent Res 100: 515-521. https://doi.org/10.1177/0022034520979133

43. Ma'an MN, Nakajima M, Foxton RM, Tagami J (2008) Bond strength and ultimate tensile strength of resin composite filled into dentine cavity; effect of bulk and incremental filling technique. J Dent 36(3): 228-234. https://doi.org/10.1016/j.jdent.2007.12.006

44. França FÁ, Oliveira MD, Rodrigues JA, Arrais CAG (2011) Pre-heated dual-cured resin cements: analysis of the degree of conversion and ultimate tensile strength. Braz Oral Res 25: 174-179. https://doi.org/10.1590/s1806-83242011000200013

45. Costa LA, Carneiro KK, Tanaka A, Lima DM, Bauer J (2014) Evaluation of pH, ultimate tensile strength, and micro-shear bond strength of two self-adhesive resin cements. Braz Oral Res 28(01): 1-7. https://doi.org/10.1590/1807-3107bor-2014.vol28.0055

46. https://www.kuraraynoritake.eu/media/pdfs/SDS_236_PANAVIA_Veneer_LC_GB_V3_2024_05_09.pdf

47. Hategekimana F, Kiraz N (2022) Preparation and characterization of silica based nanoclusters as reinforcement for dental applications. Polym Compos 43(10):7564-7574. https://doi.org/10.1002/pc.26857

48. Rodríguez HA, Kriven WM, Casanova H (2019) Development of mechanical properties in dental resin composite: Effect of filler size and filler aggregation state. Mater Sci Eng: C 101: 274-282. https://doi.org/10.1016/j.msec.2019.03.090

49. Alghazzawi TF (2024) The effects of monomer type, filler size, and filler content of three resin cements on the color stability of laminate veneers exposed to accelerated aging. Dent Mater https://doi.org/10.1016/j.dental.2024.06.001

50. Paloco EA, Berger SB, Lopes MB, Favaro JC, Gonini-Júnior A, Piauilino AI, Borba AM, Guiraldo RD (2021) Influence of resin cement and thermocycling on milled lithium disilicate ceramic microshear bond strength. Acta Odontol Latinoam 34(3):226-232. http://doi.org/10.54589/aol.34/3/226.

51. Garcia IM, Soto-Montero J, Collares FM, Giannini M (2022) Bonding of Resin Cements to Ultra-Translucent Zirconia After Aging for 24 Hours and 1 Year. Int J Prosthodont 35(4):460-468. http://doi.org/10.11607/ijp.7703.

52. Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, Dorigo EDS (2008) Dental adhesion review: aging and stability of the bonded interface. Dent Mater 24: 90-101. https://doi.org/10.1016/j.dental.2007.02.009

53. Sai K, Shimamura Y, Takamizawa et al (2016) Influence of degradation conditions on dentin bonding durability of three universal adhesives. J Dent 54: 56-61. https://doi.org/10.1016/j.jdent.2016.09.004

54. Carek A, Dukaric K, Miler H, Marovic D, Tarle Z, Par M (2022) Post-cure development of the degree of conversion and mechanical properties of dual-curing resin cements. Polymers 14: 3649.

https://doi.org/10.3390/polym14173649

55. Bolaños-Carmona V, Benavides-Reyes C, González-López S, González-Rodríguez P, Álvarez-Lloret P (2020) Influence of Spectroscopic Techniques on the Estimation of the Degree of Conversion of Bulk-fill Composites. Oper Dent 45(1):92-103. http://doi.org/10.2341/18-095-L.