Shear Bond Strength of Dentin Bonded to Various Restorative Materials Using Universal Resin Cements

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Abstract

To evaluate the shear bond strengths and failure modes of dentin bonded to various restorative materials using three contemporary universal resin cements.

Forty human third molars were longitudinally cut into 4 pieces. Ten sectioned were randomly divided into a group classified by bonding substrate materials and universal resin cement used. Two-mm diameter cylindrical rods were fabricated from five materials; Nickel-Chromium, Gold, Lithium disilicate glass ceramic, Zirconia, and Resin matrix ceramic. Three resin cements; Panavia V5, Duo-Link Universal, RelyX Universal were used to bond cylindrical specimens to prepared dentin surfaces according to the manufacturer's instructions. After subjected to thermocycling, shear bond strength was measured using a universal testing machine, and failure modes were assessed with a measuring microscope.

Lithium disilicate glass ceramic displayed the highest bond strength of 36.14 MPa when bonded with Panavia V5, while Nickel-Chromium exhibited the highest bond strength of 38.02 MPa when bonded with Duo-Link Universal. The comparable bond strength could be obtained when using these two cements bonded Nickel-Chromium, Zirconia, and Lithium disilicate glass ceramic ($p > 0.05$). While, bonding to Gold and Resin matrix ceramic provided the inferior bond strength. No significant differences were found among all substrates when RelyX Universal cement was used ($p > 0.05$). The most common failure pattern was adhesive failure between restorative material and resin cement, followed by adhesive failure between dentin and resin cement.

Gold and Resin matrix ceramic exhibited lower shear bond strength compared to the other materials, whereas Nickel-Chromium, Zirconia, and Lithium disilicate glass ceramic showed high shear bond strength when Panavia V5 and Duo-Link Universal were used. RelyX Universal showed lower shear bond strength than other cements and there were no significant differences among all substrates.

Experimental article (J Int Dent Med Res 2024; 17(1): 1-10)

Keywords: Resin cement, Dentin Bonding Agent, Dental adhesive, Primers, Shear strength.

Received date: 08 January 2024 **Accept date:** 23 February 2024

Introduction

The bonding efficiency of resin cements depends on many factors, such as substrate surface treatment or types of resin cement.[1-3] There are three types of resin cement classified by substrate modification mechanism; etch-and-rinse, self-etch, and self-adhesive resin cement. The later system is currently widely used due to its simplicity in bonding procedures. However, many researchers found that the self-adhesive resin cement provided inferior bond strength compared to other systems, especially in the case where pre-surface treatments were not achieved.[4,5]

To improve the bonding capability of resin cement to each dental substrate, different surface treatments have been established. Metal primer application was found to be effective for bonding to metal substrates.[6,7] Phosphoric acidic monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) were recommended to use as surface pretreatment for base metal alloy.[8] Sulfur-containing monomers were recommended for use as surface pretreatments for noble metal alloy.[9] Hydrofluoric

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acid etching followed by silane application improved the bond strength for the glass-based ceramic surface.[10,11] Several studies revealed that the application of MDP primer enhanced the adhesion between resin cement and zirconia surfaces as well as tooth surfaces.[12] For resin matrix ceramic, treatment with primer containing MMA was found to produce stronger bonding between resin cement and the resin matrix ceramic surface.[13,14] Due to the significant improvement of the bonding interface after surface chemical treatment, contemporary resin cement systems are presently including additional primers for application to tooth or/and restorative substrate surfaces prior to cementation. These cements are termed universal resin cement.

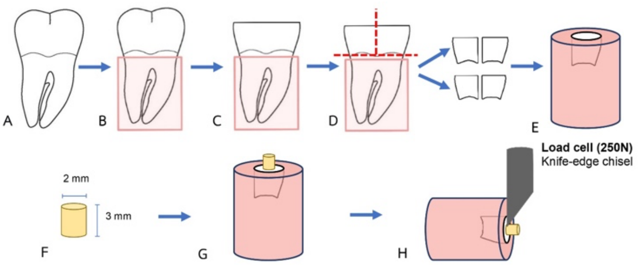

Figure 1. Specimen preparation and testing apparatus for shear bond test. A: Human third molar B: Tooth embedded in acrylic resin. C: Flat occlusal dentin surface. D: Sectioned tooth in 4-parts. E: Specimen embedded in acrylic resin. F: Cylindrical restorative material sample. G: Restorative material bonded to dentin. H: Shear bond strength testing.

Some previous studies have compared the bond strength of these new cement systems to enamel, dentin, and dental restorative substrates.[15-19] However, most studies focused on only one bonding interface between resin cement and each substrate, and generally built testing blocks with resin composite after cement application. However, bonding interfaces between dentin and various substrates using resin cement are complex.[20-22] With the improvement of resin cements and surface-treated primers, it might be questioned that which interface actually obtains inferior bond strength and initiates the failure of the restoration. Research evaluating the bond strength of dentin to various restorative material substrates using novel universal resin cement systems under aging simulation should be performed to clarify the problem of cemented restoration, including noble metal, base metal, glass ceramic, zirconia, and resin-matrix ceramic substrates. The results would be a useful clinical guideline for selecting cementation procedures for each dental restorative material.

Product	Composition	Manufacturer
Argeloy N.P.	Ni 61.2%, Cr 25.8%, Mo11.0%, Si 1.5%, Mn <1%, Al <1%	Argen Co, San Diego, USA
Aurium D69	Au 69%, Pt 4.1%, Pd 1.9%, Ag 14.5%, Cu 9.45%, Zn 1%, Ir <1%	Aurium Research, San Diego, USA
IPS e.max Press	Lithium disilicate glass ceramic	Ivoclar Vivadent, Schaan, Liechtenstein
Cercon	Zirconia ceramic	Dentsply Sirona, Bensheim, Germany
Shofu disk HC	UDMA, TEGDMA, 61 wt% silica powder, micro fumed silica, zirconium silicate	Shofu, Kyoto, Japan
IPS Ceramic Etching Gel	4.5% hydrofluoric acid gel	Ivoclar Vivadent, Schaan, Liechtenstein
Panavia V5	- Tooth primer (self-etching primer): 10-MDP, 2-HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water - Clearfil ceramic primer plus (prosthesis primer): 3-Methacryloxypropyl trimethoxysilane, 10-MDP, ethanol - Paste: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroalminosilicate glass filler, colloidal silica Bisphenol A, silanated aluminium oxide filler, dl-Camphorquinone, pigments	Kuraray Noritake Dental, Tokyo, Japan
Duo-Link Universal (Kit with All-bond universal)	- All-Bond Universal: 10-MDP, Bis-GMA, HEMA, ethanol, water, initiators - Z-prime plus: Biphenyl dimethacrylate, MDP, ethanol - Porcelain primer: Silane coupling agent - Paste: Bis-GMA, triethyleneglycol dimethacrylate, glass filler	BISCO Inc, Schaumburg, IL, USA
RelyX Universal with Scotchbond Universal Plus	- Scotchbond Universal Plus: Bisphenol A derivative free dimethacrylate monomers including a novel radiopaque monomer, MDP Phosphate Monomer, HEMA hydrophilic monomer, 3M Vitrebond Copolymer, Non-settling silica filler, ethanol, water, photo initiator, optimized mixture of silanes, dual-cure accelerator - Paste: Bisphenol A derivative free dimethacrylate monomers, phosphorylated dimethacrylate adhesion monomers, photo initiator, Novel amphiphilic redox initiator, Radiopaque fillers, rheological additives, pigments	3M ESPE, St Paul, MN, USA

MDP: Methacryloyloxydecyl dihydrogen phosphate; VBATDT: 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithiol; HEMA: Hydroxyethyl methacrylate; Bis-GMA: Bisphenol A diglycidylmethacrylate; TEGDMA: Triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate; MMA: Methyl Methacrylate; MPTMS: γ-methacryloxypropyl trimethoxysilane

Table 1. Materials, composition, and manufacturer of product used in the study.

The purpose of this study was to evaluate the shear bond strength of dentin bonded to various restorative material substrates using three contemporary universal resin cements and determine the failure mode of the bonding interfaces. The null hypotheses were that there was no significant difference in the bond strength of the dentin bonded to various restorative material substrates, and no significant difference in bond strength among the three universal resin cements used to bond dentin to various restorative material substrates.

Materials and methods

Preparation of dentin surfaces

The materials and composition used in this study are shown in Table 1. Forty extracted human third molars were collected and kept in a 0.1% thymol solution at room temperature until they were used. All teeth did not present caries, restoration, or attrition at the occlusal surface, and there was no craze line at any surface. All teeth were cleaned to remove calculus and soft tissues. The teeth were mounted up to 1 mm below
the cementoenamel junction in self-cured acrylic resin using a plastic tube.

The occlusal surface of teeth was cut to obtain a flat dentin surface. Each tooth was then cut in buccolingual and mesiodistal dimensions and cut at the cementoenamel junction to separate the occlusal surface into four pieces. The cutting procedure was performed by a specimen cutting machine with water cooling (Buehler, ISOMET 1000, Buehler Ltd., Lake Bluff, Illinois, USA). All the sectioned teeth were embedded in self-cured acrylic resin using a plastic tube with the flat dentin surface facing upward.

The dentin surfaces were finished with 600-grit abrasive paper by using a rotary grinding machine (Buehler, Metaserve, Buehler Ltd., Lake Bluff, Illinois, USA) to create a uniformly flat surface (Figure 1).

Preparation of restorative material specimens

Five types of indirect restorative materials, as shown in Table 1, thirty pieces of each, were prepared according to the manufacturer's instructions. Nickel-Chromium alloy (Argeloy N.P.), gold alloy (Aurium D69), and IPS e.max Press specimens were fabricated by conventional lost-wax and casting methods using wax patterns created by CAD/CAM techniques. Zirconia (Cercon) and resin-based ceramic (Shofu disk HC) specimens were produced by the CAD/CAM technique. The specimens were cylindrical in shape, 2 mm in diameter and 3 mm in height. The bonding surfaces of all specimens were finished with 1000-grit abrasive paper by using a rotary grinding machine (Buehler, Metaserve, Buehler Ltd., Lake Bluff, Illinois, USA) to create a uniform surface.

Restorative material	Surface treatment procedures
Nickel-Chromium alloy (NiCr)	Airborne-particle abrasion with 50 µm Al_2O_3 for 10 seconds at a distance of 10 mm, a pressure of 2.5 bar perpendicular to surface. (Sarafianou et al, 2008[6], Butler et al, 2018[20])
Gold alloy (Gold)	
Zirconia ceramic (Zr)	
Resin-matrix ceramic (RMC)	Airborne-particle abrasion with 50 µm Al_2O_3 for 10 seconds at a distance of 10 mm, a pressure of 1 bar perpendicular to surface. (Strasser et al, 2018[39])
Lithium disilicate glass ceramic (LDS)	Etching with 4.5% Hydrofluoric acid gel for 20 seconds, rinse with water spray for 5 seconds. (Manufacturer's instructions)

Table 2. Surface treatment of restorative materials before cementation.

Before cementation of indirect restorative materials to dentin surfaces, the material surfaces were treated by the procedures shown in Table 2. The specimens were then washed in an ultrasonic bath filled with distilled water for 5 minutes and air dried.

Resin cement	Substrates	Bonding procedures
Panavia V5 (PV5)	Dentin	- Wash with water spray and dry with air syringe for 10 seconds. - Apply tooth primer with agitating for 20 seconds, dry with air syringe for 5 seconds.
	Restoration	- Apply clearfil ceramic primer plus 10 seconds and dry with air syringe for 5 seconds. - Apply mixed paste to surface of restoration and place restoration on dentin, then remove excess cement with a microbrush.
Duo-link universal (Kit with All-Bond Universal) (DuL)	Dentin	- Wash with water spray, and use cotton pellets to dry it off - Apply two separate coats of All-bond universal with agitating for 15 seconds per coat. Evaporate excess solvent by strongly air-drying with air syringe for 10 seconds (no visible movement of adhesive). Light cure for 10 seconds.
	Restoration	- Apply 1 coat of Z-prime plus on metal and zirconia surface and dry with air syringe for 3 seconds. - Apply 1 coat of porcelain primer (silane) on lithium disilicate glass and resin-based ceramic surface, wait for 30 seconds, and dry with air syringe for 3 seconds. - Apply mixed paste to surface of restoration and place restoration on dentin, then remove excess cement with a microbrush.
RelyX Universal with Scotchbond Universal Plus (ReX)	Dentin	- Wash with water spray, and use cotton pellets to dry it off - No additional treatment
	Restoration	- Apply Scotchbond Universal Plus 20 seconds and dry with air syringe for 5 seconds. - Apply mixed paste to surface of restoration and place restoration on dentin, then remove excess cement with a microbrush.

Table 3. Bonding procedures.

Cementation procedures

Three resin cements, Panavia V5 (Kuraray Noritake Dental, Tokyo, Japan), Duo-Link Universal (BISCO Inc, Schaumburg, IL, USA), and RelyX Universal (3M ESPE, St Paul, MN, USA) were used to bond each of the restorative materials to dentin surface. The cements were handled according to the manufacturer's instructions, which are summarized in Table 3.

After completing chemical surface treatment on each surface, the mixture of resin cement was applied to the restorative material substrates, which were then boned to the dentin surface under a pressure of 750 grams for 10 minutes (Gillmore apparatus). Light-curing was performed for 20 seconds each from 4 directions by placing the tip of the LED light curing (Elipar Trilight; 3M ESPE, St Paul, MN, USA) close to the bonding interfaces. A light intensity of 800 mw/cm2 was used and calibrated by a radiometer (Coltolux, Coltene Whaledent, Inc., Ohio, US) to ensure maximum polymerization of resin cements.[23,24]

All the bonded specimens were stored in distilled water at 37°C for 24 hours, allowing complete polymerization. The bonded specimens were then subjected to thermocycling between 5°C and 55°C for 5000 cycles with a 30 second dwell time.

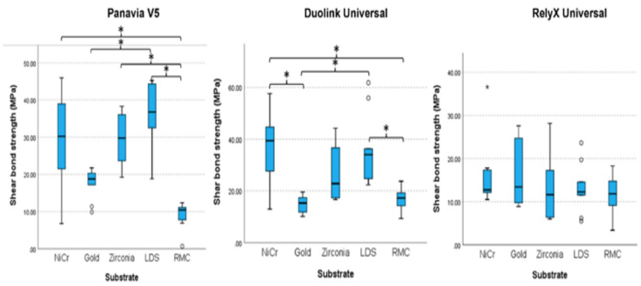

Figure 2. The median of each experimental group when comparing within the same cement A. Panavia V5; B. Duolink Universal; C. RelyX Universal; The asterise (*) indicates significant difference (p < 0.05).

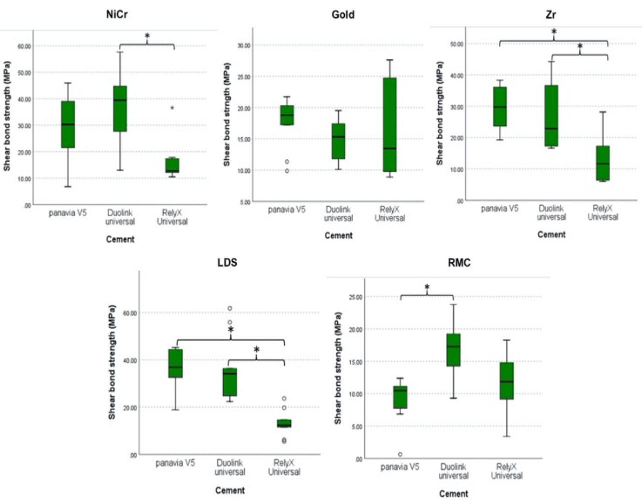

Figure 3. The median of each experimental group when comparing within the same substrate. A. Nickel-Chromium alloy; B. Gold alloy; C. Zirconia ceramic; D. Lithium disilicate glass ceramic; E. Resin-matrix ceramic; The asterise (*) indicates significant difference (p < 0.05).

Shear bond strength test

After thermocycling, the shear bond strength test was carried out on a universal testing machine (Lloyd instruments, Model LRX-Plus, AMETEK Lloyd Instrument Ltd., Hamphshire, UK). All specimens were loaded to failure (Fig.1) at a crosshead speed of 0.5 mm/min. using a knife-edge chisel that was placed close to the bonding interface. The maximum failure load was recorded in Newton (N). The shear bond strength values of each specimen were calculated in MPa by dividing the failure load (N) by the bonding area (mm^2), which was πr^2; where r is the radius of the bonding interface, 1 mm.

Cement / Substrate	Panavia V5	Duolink Universal	RelyX Universal
NiCr	28.62 (11.97)^{AB,ab}	38.02 (13.79)^{A,a}	16.03 (7.73)^{A,b}
Gold	17.50 (3.88)^{BC,a}	14.79 (3.59)^{B,a}	16.11 (7.48)^{A,a}
Zirconia	29.44 (6.98)^{AB,a}	27.07 (11.06)^{AB,a}	12.76 (7.10)^{A,b}
LDS	36.14 (8.42)^{A,a}	36.47 (13.87)^{A,a}	13.10 (5.79)^{A,b}
RMC	8.96 (3.60)^{C,a}	16.85 (4.76)^{B,b}	11.64 (4.55)^{A,ab}

Table 4. Mean shear bond strength values (standard deviations) (MPa) for all groups.
Different uppercase letters indicate significant differences in columns. Different lowercase letters indicate significant differences in rows (Kruskal Wallis and Bonferroni correction, $p < 0.05$). Abbreviation: NiCr=Nickel-Chromium, LDS=Lithium disilicate, RMC = Resin matrix ceramic.

Determine the mode of failure

After shear bond strength testing, the failure mode was assessed by inspecting the bonding surfaces of each specimen under a measuring microscope (Nikon, MM-400/L, Nikon Corporation, Tokyo, Japan). The percentages of each failure mode were defined by the ImageJ software. The failure mode was classified into one of the following eight groups as follow: 1. Adhesive failure between dentin and resin cement (ADC) 2. Adhesive failure between restorative material and resin cement (ASC) 3. Cohesive failure in dentin (CD) 4. Cohesive failure in resin cement (CC) 5. Cohesive failure in restorative material (CS) 6. Mixed failure at the resin cement-dentin interface (MCD) 7. Mixed failure at the resin cement-restorative material interface (MCS) 8. Mixed failure at the resin cement-restorative material interface and the resin cement-dentin interface (MA). The criteria to determine each failure mode are as follows: Adhesive failure occurs when failure affects equal to or more than 70% of the entirely bonded area within the bonding interface (whether it's the dentin-resin cement interface or the restorative material-resin cement interface). Cohesive failure takes place when failure affects equal to or more than 70% of the entirely bonded area within the substrate (be it dentin, resin cement, or restorative material). Mixed failure refers to a combination of adhesive and cohesive failure within a single bonding interface (such as the resin cement-dentin interface or the resin cement-restorative material interface).

Substrates (S)	Adhesive resin cement	Mode of failures (n)							
		CC	CD	CS	ASC	ADC	MA	MCS	MCD
NiCr	PanaviaV5	3	0	0	0	0	0	5	2
	Duolink Universal	2	0	0	5	0	1	2	0
	RelyX universal	0	0	0	0	9	0	0	1
Gold	PanaviaV5	0	0	0	9	1	0	0	0
	Duolink Universal	0	0	0	10	0	0	0	0
	RelyX universal	0	0	0	8	0	2	0	0
Zr	PanaviaV5	0	0	0	7	1	2	0	0
	Duolink Universal	0	0	0	7	0	0	3	0
	RelyX universal	0	0	0	0	10	0	0	0
LDS	PanaviaV5	8	0	0	0	2	0	0	0
	Duolink Universal	0	0	0	9	1	0	0	0
	RelyX universal	0	0	0	8	0	2	0	0
RMC	PanaviaV5	0	0	0	9	1	0	0	0
	Duolink Universal	0	0	0	5	0	0	5	0
	RelyX universal	0	0	0	0	8	0	2	0
Total (%)		9	0	0	51	22	5	11	2

Table 5. Number of specimens in each failure mode.

SEM observation of the fractured surfaces

The failed specimens were selected for scanning electron microscope observation (HITACHI SU3900, Tokyo, Japan). The specimens were gold sputtered by a sputter coater (SPI-MODULE, Laughton, East Sussex, UK). Representative photomicrograph using 40x, 500x, 1,000x magnifications were taken to represent characteristics of each failure mode.

Statistical analyses

The data from shear bond strength tests were analyzed for normal distribution using the Kolmogorov-Smirnov test and homogeneity of variance using Levene’s test. A non-normal distribution of collected data in each experimental group was attained. The data were analyzed with the Kruskal Wallis test and followed by pair-wise comparisons with Dunn’s post hoc test. The bonferroni correction was used to compare the differences between each experimental group. Statistical testing was performed at a 95% level of confidence ($\alpha = .05$).

Results

The mean shear bond strength and standard deviations for all experimental groups are presented in Table 4. The median of the bond strength values and the significant differences ($p < 0.05$) are shown in Figure 2 and 3.

When bonding the substrates to dentin with PV5, LDS exhibited the highest mean shear bond strength (36.14 MPa). Zr and NiCr provided similar bond strengths of 29.44 and 28.62 MPa, respectively. There was no significant difference in bond strength values among these three materials. While the bond strength of the gold alloy, was 17.5 MPa, which was significantly lower than that of LDS. However, statistical comparison revealed no significant difference between Gold and Zr and NiCr, although the mean bond strength values were approximately 10 MPa lower. The bond strength of RMC was only 8.96 MPa, and it was definitely lower than those of the other materials ($p<0.05$), except Gold. When using DuL, NiCr demonstrated the highest mean shear bond strength to dentin (38.02 MPa). However, no significant difference was observed between Zr (27.07 MPa) and LDS (36.47 MPa). In contrast, Gold displayed the weakest shear bond strength to dentin with bond strength value of 14.79 MPa, which was not significant different from Zr (27.07 MPa) and RMC (16.85 MPa). Surprisingly, there were no significant differences among all substrates when bonding dentin surfaces with ReX ($p>0.05$). The bond strengths ranged from 11.64 MPa – 16.11 MPa for five substrates.

When comparing within the same substrate, DuL demonstrated the highest mean shear bond strength for NiCr (38.02 MPa), and there was no significant difference to that of PV5 (28.62 MPa). Conversely, ReX exhibited the lowest shear bond strength (16.03 MPa), with no discernible difference from PV5. There was no significant difference observed among all cements when bonding to Gold ($p>0.05$). The bond strength values were approximate at only 14.79 - 17.50 MPa. Regarding Zr and LDS, there was a similar trend in bond strength values. PV5 and DuL provide the comparable bond strength ($P>0.05$), while the bond strength obtained from

ReX was significantly lower than those of the two cements. For RMC, DuL demonstrated the highest mean shear bond strength to dentin (16.85 MPa), which was no significant difference from that of ReX (11.64 MPa). Conversely, PV5 exhibited the lowest mean shear bond strength (8.96 MPa) with no significant difference from that of ReX.

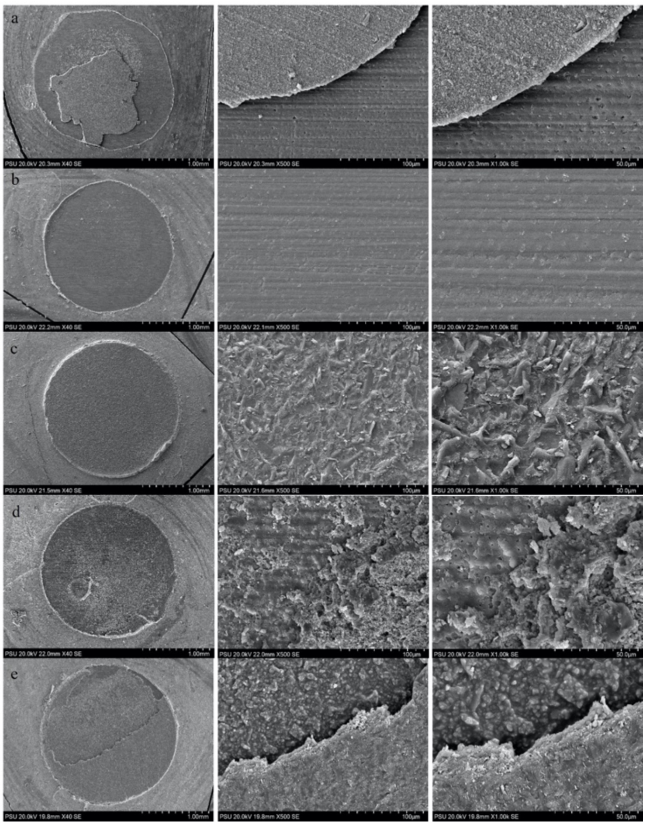

Figure 4. a) Scanning electron microscope images (×40, ×500, ×1000 magnification) in dentin site luting between LDS and ReX showing mixed failure at resin cement-restorative material interface and resin cement-dentin interface. Abrasive polished surface of dentin was observed at resin cement-dentin interface.
b) Scanning electron microscope images (×40, ×500, ×1000 magnification) in dentin site luting between Zr and ReX showing adhesive failure between dentin and resin cement. Abrasive polished surface of dentin was predominantly detected.
c) Scanning electron microscope images (×40, ×500, ×1000 magnification) in dentin site luting between Gold and PV5 showing adhesive failure between restorative material and resin cement. The resin particles covered the dentin surface, and abrasive polished surface of dentin was not observed.
d) Scanning electron microscope images (×40, ×500, ×1000 magnification) in dentin site luting between NiCr and PV5 showing mixed failure at resin cement-dentin interface. Abrasive polished surface of dentin and resin cement cluster were simultaneously observed.
e) Scanning electron microscope images (×40, ×500, ×1000 magnification) in dentin site luting between NiCr and PV5 showing mixed failure at resin cement-restorative material interface. Resin cement cluster and substrate surface were simultaneously noticed.

For failure mode observation, the frequency distribution of failure modes is presented in Table 5. Representative scanning electron microscope (SEM) images in each failure mode are displayed in Figure 4 For precious metal, Gold, the failure patterns were mainly occurred at the interface between cement and substrate interface regardless of the cement type. On the other hand, the base metal alloy (NiCr) and RMC demonstrated various type of failure mode depending on the cement used. Cohesive failure in cement including the mixed mode of this failure type frequently observed when PV5 was used. Adhesive failure between substrate and cement was 50% detected for DuL. Almost all of the specimens bonded with ReX failed as adhesive failure between dentin and cement, except for Gold and LDS failed as adhesive failure between cement and substrate. The Zr interfaces predominantly failed as adhesive failure between cement and substrate for PV5 and DuL whereas adhesive failure between dentin and cement occurred for ReX.

Discussion

The present study evaluated the shear bond strength of dentin bonded to various restorative material substrates using three universal resin cements (PV5, DuL, and ReX) with the main purpose for defining the bonding strength level and failure site of each material substrate bonded to the dentin surface. Statistical analysis indicated that, except for the gold alloy surface, the shear bond strength of dentin bonded to various restorative material substrates was affected by the types of universal resin cement. The null hypothesis was rejected. In addition, the bond strength of PV5 and DuL were affected by the type of substrate materials.

Thermocycling is the condition most often used to test the durability of resin bonds. This is considered a clinically relevant aging parameter. It was found that thermocycling played a significant role in some resin cements degradation, leading to significantly lower shear bond strength. Several studies reported that thermocycling significantly decreased the bond strength of self-etch and self-adhesive resin cements.[18,25,26] Therefore, all specimens in the present study were subjected to thermocycling to simulate aging condition to provide more clinically relevant results.

Statistical comparison in some groups revealed no significant difference, even though the mean bond strength values appeared distinct. The bond strength test is a sensitive method. A defect in the bonding interface generates non-uniform stress distribution in the defective area, which then initiates crack formation.[27] The large variation in bond strength values created a non-normal data distribution, a non-parametric statistical analysis was therefore employed in the present study. Consequently, the median was utilized instead of the mean, resulting in the removal of data outliers. As a result, the significant difference was difficult to detect.

The shear bond strength tests were performed in several studies since no further specimen processing is needed after the bonding procedure. However, cohesive failures in the substrate were frequently observed with new adhesives that obtained better bond strength. The previous study indicated that small surface areas of test specimens were associated with higher bond strengths. Nevertheless, micro-shear bond strength testing is a sensitive technique in fabricating specimens due to a very small size of 1-mm diameter specimens. For this reason, a small size shear specimen was proposed in this study to reduce technique sensitivity and the number of defects in the bonding interface, leading to a reduction in cohesive failure. Previous studies recommended the retentive area in cylindrical shape range from 3 to 7 mm^2 (2 to 3 mm in diameter).[15,20] In the present study, the retentive area had a diameter of 2 mm. It was observed that the predominant failure mode was adhesive failure, suggesting that the methods used are acceptable.

Regarding the type of material, LDS bonded using PV5 showed the highest mean shear bond strength (36.14 ± 8.42 MPa). The predominant failure pattern was cohesive failure within the cement, indicating that the cement bonded to the dentin surface as effectively as to the LDS surface. While DuL exhibited the highest mean shear bond strength to the NiCr (38.02 ± 13.79 MPa). Adhesive failure between the substrate and cement was detected at a rate of 50%, suggesting that the cement bonded more effectively to the dentin surface than to the NiCr surface. It was found that no significant difference in bond strength was observed for LDS, NiCr, and Zr surfaces (ranging from 27.07 MPa - 38.02 MPa) when they were bonded with these two cements. Whereas similar bond strengths were obtained for all substrates when ReX was used. PV5 and DuL are composed of tooth primers, which contain 10-MDP and restorative primers, which contain 10-MDP and silane.

The monomer 10-MDP readily adhered to hydroxyapatite. This bond appeared very stable, as confirmed by the low dissolution rate of its calcium salt in water.[28] Therefore, several current resin cement systems include MDP in separate primers or in the resin itself to improve dentin bonding quality. 10-MDP also promoted bond strength in base metal alloy and zirconia.[8,29] While silane coupling agent provide a chemical covalent and hydrogen bond, which was a major factor for a sufficient resin bond to silica-based ceramics.[11] The results of the present study are in agreement with those of previous studies. It was demonstrated that 10-MDP promoted bond strength in NiCr and Zr. Shafiei et al showed that sandblasting and metal primers containing MDP improved the shear bond strength of both tested resin cements to NiCr.[15] In addition, it was found that sandblasting and priming with Z-Prime plus improved the shear bond strength of Zr when using DuL.[17] Many researchers proved that MDP-containing primers applied to the Zr surface before cementation were more essential to increase the bond compared to MDP-containing resin cement.[30-32]

For LDS, a strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface. Acid etching with solutions of hydrofluoric acid (HF) was found to be an effective method for treating ceramic surfaces to obtain micromechanical retention.[10] HF attacks the glass phase of glass-matrix ceramics, causing micro-retention due to dislodgement of the crystal and achieving proper surface texture and roughness. Using of 5% HF for 20 seconds or 2.5% HF for 120 seconds was effective enough to properly dissolve the glassy matrix and achieve greater bond strength.[33]

Silane coupling agents provide a chemical covalent and hydrogen bond, which is a major factor in providing a sufficient resin bond to silica-based ceramics. Etching with HF acid alone was not sufficient to produce a strong bond with dental ceramics, but when a silane coupling agent was used, the adhesion bond increased.[34] Therefore, it could be noticed from the present results that PV5 and DuL can promote better bond strength for NiCr, Zr, and LDS. Especially for LDS, the bond strength could be as high as 36 MPa for both cements. The predominant failure pattern for these two cements was adhesive failure between the substrate and cement, except in the case of PV5 to LDS, where there was a cohesive failure within the cement. It is noteworthy that their tooth primers were more effective for dentin surface bonding compared to the substrate. In the case of Gold and RMC, the bond strength tended to be lower compared to the other materials. However, no significant difference was observed in the shear bond strength between these two substrates. The most common failure pattern, similar to other substrates, was adhesive failure between the substrate and cement. The previous studies showed that the primer containing VBATDT will promote a strong and durable bond to noble alloys.[35] The primer containing MMA was found to produce stronger bonding to resin-based CAD/CAM materials.[13,14] It could be noticed that PV5 and DuL do not contain these two specific monomers, resulting in lower bond strengths for Gold and RMC compared to the other substrates. Several studies revealed that the adhesion of resin cement to noble metal surfaces were very weak without a specific primer.[9,35] RMC are composed predominantly (>50% by weight) of refractory inorganic compounds. Previous studies compared the shear bond strengths of CAD/CAM composite blocks and composite cements using different primers and found that a primer containing MMA (HC primer) exhibited significantly higher bond strength to CAD/CAM composite blocks.[13,14,36] Further study should be performed to evaluate the shear bond strength of these two substrate materials when used together with those specific primers.

In comparison to other cements, ReX provided inferior shear bond strength, with no discernible difference seen across all substrates. Contrary to the other two cements, which are self-etch resin cements composed of a separating primer for tooth and substrate surface. ReX is a self-adhesive resin cement that does not require a primer for the tooth, it only includes a single-bottle universal bonding agent for substrate surface treatment. All cements in this study were used according to the manufacturer's instructions. Therefore, the dentin surface in the self-adhesive resin cement group (ReX) did not receive any pre-surface treatment, which differed from the other two cements. Its bonding quality depends on functional acidic monomers in its components, without the need for a separating primer for dentin. The separating acidic primer would enhance dentin wettability before applying the cement paste. However, Scotchbond Universal Plus, a universal primer for ReX, was applied only to the substrate surface and not for dentin pretreatment. This present study reported that a self-adhesive resin cement (ReX) exhibited lower shear bond strength compared to self-etch resin cements (PV5 and DuL). This result was consistent with previous studies.[21] Several studies reported that etch-and-rinse and self-etch resin cements had significantly higher bond strengths than self-adhesive resin cements.[4,5,21,37] The weak adhesion at the cement-dentin interfaces was confirmed by the predominant failure mode, which primarily occurs as adhesive failure between dentin and resin cement when ReX was used.

When comparing the bond strength among three cements within the same substrate, it was found that there were no significant differences among the cements when bonding to Gold (ranging from 14.79 MPa – 17.50 MPa). This was because the additional primers in these three cements do not contain a specific primer for Gold, such as VBATDT, as mentioned above. For NiCr, DuL showed the highest mean shear bond strength (38.02 ± 13.79 MPa), and there was no significant difference compared to PV5 (28.62 ± 11.97 MPa). These two cements have separate restorative primers that contain 10-MDP, which is specific for NiCr[8]. The 10-MDP primer of PV5 and DuL was also the reason for the high bond strength of Zr. Although Gold exhibited rougher surface than Zr

(Fig.4b,4c) but the chemical bond was not as strong as Zr. For LDS, separated ceramic primers (silane coupling agent) were utilized for PV5 and DuL. High bond strengths (36 MPa) were obtained for both cements. Unfortunately, the ReX system does not include a separate ceramic primer. Scotchbond Universal Plus, a universal primer for ReX, contains a mixture of various agents, including silane, dimethacrylate monomer, MDP phosphate monomer, and others, which results in a lower quantity of silane compared to the other two cements that have pure silane in their separating primers. Therefore, the bond strength of ReX was relatively low. In the case of RMC, DuL showed the highest mean shear bond strength (16.85 ± 4.76 MPa), while PV5 showed the lowest mean shear bond strength (8.96 ± 3.60 MPa). However, there was no significant difference in ReX. This can be explained by the fact that PV5 has only one bottle of a restorative primer, which is a mixture of MDP and silane, whereas DuL has two separate restorative primers that isolate MDP and silane. Therefore, the silane coupling agent in DuL exhibited greater efficacy compared to that in PV5. In the present study, Shofu HC is zirconia-silica ceramic in a resin interpenetrating matrix. The preceding systematic review and meta-analysis revealed that sandblasting had been advocated as the preferred pretreatment for CAD/CAM hybrid ceramics with high ceramic content, such as Shofu HC. Conversely, pre-treatment with hydrofluoric acid (HF) is recommended for CAD/CAM resin nanoceramics and glass ceramic in a resin interpenetrating matrix, such as Lava Ultimate or Vita Enamic. Nevertheless, both methods were followed by the application of silane to achieve enhanced bond strength.[38]

Conclusions

Within the limitations of this study, it can be concluded that the dentin shear bond strength was affected by the types of universal resin cement and substrate materials. Gold and RMC exhibited lower shear bond strength compared to the other materials, whereas NiCr, Zr, and LDS showed similar high shear bond strength when PV5 and DuL were used. ReX showed lower shear bond strength than other cements, and there were no significant differences among all substrates. The failure patterns mainly occurred at the interface between cement and substrate. Almost all of the specimens bonded with ReX failed as adhesive failure between dentin and cement.

Acknowledgements

The authors gratefully acknowledge the financial support received from the Thailand Research Fund (TRF) and the Commission on Higher Education, Thailand.

Declaration of Interest

The authors report no conflict of interest.

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