

RESEARCH ARTICLE

A comprehensive in vitro study on the performance of two different strategies to simplify adhesive bonding

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Abstract

Objective: The purpose of this study is to compare the bonding performance and mechanical properties of two different resin composite cements using simplified adhesive bonding strategies.

Materials and methods: Shear bond strength of two resin composite cements (an adhesive cement: Panavia V5 [PV5] and a self-adhesive cement: RelyX Universal [RUV]) to human enamel, dentin, and a variety of restorative materials (microfilled composite, composite, polymer-infiltrated ceramic, feldspar ceramic, lithium disilicate and zirconia) was measured. Thermocycle aging was performed with selected material combinations.

Results: For both cements, the highest shear bond strength to dentin was achieved when using a primer (PV5: 18.0 ± 4.2 MPa, RUV: 18.2 ± 3.3 MPa). Additional etching of dentin reduced bond strength for RUV (12.5 ± 4.9 MPa). On enamel, PV5 achieved the highest bond strength when the primer was used (18.0 ± 3.1 MPa), while for RUV etching of enamel and priming provided best results (21.2 ± 6.6 MPa). Shear bond strength of RUV to restorative materials was superior to PV5. Bonding to resin-based materials was predominantly observed for RUV.

Conclusions: While use of RUV with the selective-etch technique is slightly more labor intensive than PV5, RUV (with its universal primer) displayed a high-bonding potential to all tested restorative materials, especially to resin.

Clinical significance: For a strong adhesion to the tooth substrate, PV5 (with its tooth primer) is to be preferred because etching with phosphoric acid is not required. However, when using a wide range of varying restorative materials, RUV with its universal primer seems to be an adequate option.

KEYWORDS

ceramic primer, MDP, Panavia V5, RelyX universal, resin composite cement, shear bond strength, thermocycle aging, universal primer

1 | INTRODUCTION

The longevity of indirect restorations strongly relies on the bond strength that the resin composite cement achieves and maintains to its substrates.¹ Adhesion between dentin and resin composite cement is generally more prone to failure than bonding to enamel or silicate ceramic.^{1,2} Furthermore, composites, and zirconia are considered challenging substrates for achieving a long-term chemical bond.^{3–5}

The micromechanical bond of resin composite cement to dentin is based on the infiltration and polymerization of small monomers into the collagen fibril network, referred to as hybrid layer.^{6,7} This layer not only seals dentin, but also prevents post-operative sensitivity, secondary caries, and acts as an elastic buffer compensating for tensile stress generated by polymerization shrinkage of resin composite cement.^{7–9}

To achieve a hybrid layer using the traditional etch-and-rinse approach, dentin is pretreated with an acidic agent (maleic acid or phosphoric acid), with subsequent application of hydrophilic monomers penetrating into dentin tubules. The primer is then coated with a more hydrophobic co-monomer mixture (adhesive), polymerized, and the resin cement is placed over that. Although this multi-step adhesion technique has been successfully applied for composite fillings for decades, it is rather time-consuming in clinical practice.

With the wide range of CAD/CAM materials now available that require different surface treatments to achieve long-term bonding, knowledge of material properties is paramount.^{10–12} Silicate ceramics are commonly pretreated by etching with hydrofluoric acid to increase the bondable surface area and to generate micro-undercuts to provide interlocking between substrate and cement. Chemical bonding to silicate ceramics is obtained by the application of a silane.^{13,14} Zirconia is air-abraded using alumina followed by application of a primer (10-methacryloyloxydecyl dihydrogen phosphate [MDP]) to generate chemical bonding.^{15–18} Treatment of CAD/CAM polymer materials and composites strongly relies on their specific composition. This treatment usually consists of alumina air abrasion followed by application of methacrylates that can covalently bond to the resin matrix.^{19–21}

To simplify the adhesive cementation process in clinical practice, manufacturers have developed specific approaches. Self-etch primers containing non-rinsing acidic monomers such as MDP, which can simultaneously etch and prime the tooth substrate, have been developed.^{22–26} Hence, separate etching of the tooth substrate with phosphoric acid can be omitted and a single primer bottle is required.^{27,28} To bond to restorative materials, silane and MDP are often incorporated in a ceramic primer such that a chemical bond to both silicate ceramics and zirconia is formed, respectively.^{29,30} Furthermore, use of “universal primers” containing a wide selection of components designed to specifically bond to both tooth substrate and restorative materials has increased.^{31–33} However, it remains unknown if unreacted components may leach over time and weaken the bonding performance or affect biocompatibility.^{34–36} Self-adhesive resin composite cements include monomers having phosphoric acid groups. These agents are used without a separate

adhesive system. Bonding performance of self-adhesive resin composite cements may be lower than for adhesive resin composite cements using a separate etching step because penetration into dentin tubules and micromechanical retention is limited due to the cements' viscous consistency and ability to demineralize tooth structure.²⁷ However, the clinical application of self-adhesive resin composite cements is considerably simplified compared with other cements, thus reducing the risk of handling errors and making them more attractive to clinicians.

Despite manufacturers' efforts to develop user-friendly cementation products, the question remains whether clinicians should consider using an “adhesive resin cement” system requiring separate tooth and restorative material primers or should a “self-etch cement” system be used incorporating use of a universal priming system reactive on both tooth and restorative substrates. The former type of cementation system is represented by the Panavia V5 (PV5) product line (Kuraray Noritake, Okayama, Japan).^{27,37,38} This cement requires a separate primer component to bond to tooth tissues (PTP, PV5 Tooth Primer) and a different primer to be used on restorative substrates (PCP; Clearfil Ceramic Primer Plus, Kuraray Noritake). PTP is a self-etching primer containing MDP, hence no additional pretreatment with phosphoric acid is required according to the manufacturer. PCP contains silane to bond to silicate ceramics and MDP for the pretreatment of zirconia. An example of the latter type of system (a self-etching resin cement) is the product line of RelyX Universal (RUV) (3 M; Neuss, Germany) using the “all-in-one” universal Scotchbond Universal Adhesive Plus (SBU) to be used on both tooth and restorative substrates.

The purpose of this study was to compare the bonding performance of these representative classifications of contemporary popular resin-based cementation systems to both dentin and enamel specimens of extracted human teeth. In addition, bonding abilities of the two systems to a wide variety of commonly encountered restorative substrates was measured and compared. Lastly, compressive and diametral strength tests of the two different systems were obtained and compared.

The research hypotheses tested were: (1) The shear bond strength values to tooth tissues and to selected restorative material substrates will be significantly higher when using the control, adhesive resin cement (PV5) compared with the newly introduced self-etching cement (RUV), and (2) the compressive and diametral tensile strength of the control cement (PV5) will be significantly higher compared with those of the newly introduced cement (RUV).

2 | MATERIALS AND METHODS

Shear bond strengths of the two representative resin composite cement systems (PV5, RUV) to human enamel and dentin and to a wide variety of restorative materials were investigated. Additionally, the compressive and diametral tensile strengths of the cement materials were evaluated. All materials used are presented in Table 1.

TABLE 1 Material compositions obtained from manufacturer's information and safety data sheets

Type	Name	Code	Manufacturer	Composition
Cement				
Adhesive resin composite cement	Panavia V5	PV5	Kuraray Noritake, Okayama, Japan	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica, silanated aluminum oxide filler, di-camphorquinone, initiators, accelerators, pigments
(Self-) adhesive resin composite cement	RelyX Universal	RUV	3 M, Neuss, Germany	HEMA, UDMA, TEGDMA, phosphorylated dimethacrylate adhesion monomers, ytterbium trifluoride, glass powder surface modified, silane, trimethoxyoctyl-hydrolysis products with silica, triphenyl phosphite, t-amyl hydroperoxide, 26-di-tert-butyl-P-cresol
Primer				
Ceramic primer	Clearfil Ceramic Primer Plus	PCP	Kuraray Noritake, Okayama, Japan	3-Methacryloxypropyl trimethoxysilane, MDP, ethanol
Tooth primer	Panavia V5 Tooth Primer	PTP	Kuraray Noritake, Okayama, Japan	MDP, HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water
Universal primer	Scotchbond Universal Adhesive Plus	SBU	3 M, Neuss, Germany	Dimethacrylate monomers, MDP, HEMA, copolymer of acrylic and itaconic acid, (3-aminopropyl)triethoxysilane, silica filler, camphorquinone, N,N-dimethylbenzocaine, acetic acid, ethanol, water
Restorative materials				
Microfilled composite	CAD-Temp	CT	Vita, Bad Säckingen, Germany	14 wt% inorganic filler, 86 wt% acrylate polymer
Highly filled composite	Lava Ultimate	LU	3 M, Neuss, Germany	80 wt% inorganic nano-filler, 20 wt% resin matrix
Polymer-infiltrated ceramic	Vita Enamic	VE	Vita, Bad Säckingen, Germany	14 wt% TEGDMA/UDMA, 86 wt% feldspar ceramic
Feldspar ceramic	Vitablocs Mark II	VM	Vita, Bad Säckingen, Germany	56–64 wt% SiO ₂ , 20–23 wt% Al ₂ O ₃ , 6–9 wt% Na ₂ O, 6–8 wt% K ₂ O
Lithium disilicate	IPS e.max CAD	EC	Ivoclar Vivadent, Schaan, Liechtenstein	57–80 wt%, SiO ₂ , 11–19 wt%, Li ₂ O, 0–13 wt% K ₂ O
Zirconia	Vita YZ T	YZ	Vita, Bad Säckingen, Germany	90–95 wt% ZrO ₂ , 4–6 wt% Y ₂ O ₃ , 1–3 wt% HfO ₂ , 0–1 wt% Al ₂ O ₃ (3Y-TZP)

Abbreviations: Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

2.1 | Cement strength characterization

To evaluate the mechanical properties of the cements, the compressive and diametral tensile strengths^{39–43} of both cements were measured after auto-polymerization or after light-curing. Forty-eight cylindrical specimens having a diameter and height of 3.2 mm was produced from each cement using a Teflon mold with mylar foil and glass slides fixed from both sides.^{41,44}

Half of the specimens were allowed to auto-polymerize in the dark at 37°C and the remainder were light-polymerized for 20 s from each side (Elipar DeepCure-S, 3 M). After removal from the

mold, specimens were stored in water at 37°C for 24 h. Compressive strength (loading parallel to the cylinder axis) and diametral tensile strength (loading perpendicular to the cylinder axis) were measured using a universal testing machine (Z020; Zwick/Roell, Ulm, Germany) at a crosshead speed of 1 mm/min. Compressive and diametral tensile strength were calculated using the following formula:^{41,44}

$$\text{Compressive strength: } \sigma = F/\pi(d/2)^2.$$

$$\text{Indirect tensile strength } \sigma = 2F/\pi dh.$$

F is the fracture load, d is the specimen diameter, and h is the specimen height.

2.2 | Shear bond strength

Shear bond strength of the two cement systems to human enamel and dentin ($n = 15$ per group) and to restorative materials ($n = 12$ per group) was evaluated using the Swiss shear test design.^{3,27,45–48} Sample sizes were chosen according to data from previously conducted experiments^{3,27,38,45} considering an α of 0.05 and power of 0.8. Figure 1 shows the different groups and pretreatments. Material compositions are summarized in Table 1.

The use of human teeth for laboratory studies was approved by the Ethics Committee Northwest/Central Switzerland (EKNZ UBE-15/111). Intact, unrestored human third molar teeth were cleaned from any remnant tissue or contamination and stored at room temperature in an aqueous solution of 0.5% Chloramine T to minimize microbial growth. All teeth were tested less than 6 months after extraction. Teeth were embedded in a square silicon mold containing cold-curing resin (Demotec 20; Nidderau, Germany) and then wet-ground using a series of silicon carbide abrasive surfaces (Struers, Ballerup, Denmark P180, 400, 800, 1200) in a polishing machine (Minitech 265, Presi, Hagen, Germany) from the buccal or lingual side respectively to expose a flat dentin or enamel surface that was at least 5 mm in diameter. Tooth surfaces were then rinsed with water, slightly dried using oil-free air, and were then pretreated to one of the various protocols listed in Figure 1. For the etching procedure, a 35% phosphoric acid gel (Ultradent, South Jordan, UT, USA) was applied for 15 s.

The restorative materials were selected to cover the range of material types commonly used for restorations in dentistry (Table 1). CAD/CAM material blocks of representative products were sliced into discs of 2 mm thickness and wet-ground using a series of silicon carbide abrasive papers (P180, P400, P800, and P1200 [Struers]) to attain flat, parallel surfaces: CT (Vita CAD-Temp, Vita, Bad Säckingen, Germany), LU (Lava Ultimate, 3 M, Neuss, Germany), VE (Vita Enamic, Vita), VM (Vitablocs Mark II, Vita) and EC (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein). The lithium disilicate product EC was ceramed afterwards (Programat, Ivoclar Vivadent) according to the manufacturer's instructions. The zirconia-based material YZ (Vita YZ-T, Vita) was over-dimensionally milled and then ground using silicon carbide abrasive paper (final polish of P2500 grit), and sintered (Zyrcomat 6100 MS, Vita) according to manufacturer's instructions.

The restorative material surfaces to be tested were treated according to methods displayed in Figure 1. To micro-structure (air-abrasion or acid-etching) the surfaces according to the manufacturer's recommendations, alumina particle abrasion of CT, LU, and YZ was performed using 50 μm alumina particles applied with 2 bar pressure (Dento-prep, Ronvig Dental, Daugard, Denmark). Specimens were then cleaned in an ultrasonic bath (TPC-15; Telsonic AG, Bronschhofen Switzerland) for 4 min in 70% ethanol. Hydrofluoric acid (5%, Ceramics Etch, Vita) was used to etch VE (60 s), VM (60 s), and EC (20 s) and was subsequently rinsed off with water. To distinguish the effect of surface smoothness (indirect indicator of potential chemical bonding), additional specimens of all restorative materials were wet-polished using a series of silicon carbide abrasive papers (P400, P800, P1200, P2500 [Struers]), and finally using a 3- μm diamond paste. Where applicable, the respective primer was applied using a micro-brush and dried using oil-free air.

All pretreated specimens were then fixed in a customized holding device (Figure 2). An acrylic cylinder (D + R Tec, Birmensdorf, Switzerland) having an inner diameter of 2.9 mm and outer diameter of 3.1 mm was fastened vertically on the pretreated substrate surface. Either type of resin cement was applied through the opening of the acrylic cylinder onto the substrate surface. The cement was then compressed using a headless steel screw to a force of 1 N and then light-activated (Elipar DeepCure-S, 3 M) for 20 s from three different directions. Subsequently, all specimens were carefully removed from the holding device and dark-stored in water at 37°C for 24 h. Specimens that underwent thermocycle aging (aging groups according to Figure 1) were subjected to 20,000 cycles between 5 and 55°C with a dwell time of 30 s (Thermocycler THE-1100, SD Mechatronik GmbH, Feldkirchen, Germany). The material combinations for the aging group were chosen based on their clinical relevance. For the control (adhesive cement PV5) it is known from an earlier investigation,²⁷ that the application of the associated primer (PTP) without previous etching is clinically easy to handle and provides sufficient bond strength on enamel and dentin. Therefore, this procedure was chosen for the aging test. For the test material (self-adhesive cement RUV) it was decided to use the combinations with the highest initial bond strength for the aging test. The restorative materials tested after aging were CT, EC, and YZ after micro-structuring and primer application. These materials were chosen because EC and YZ are frequently used

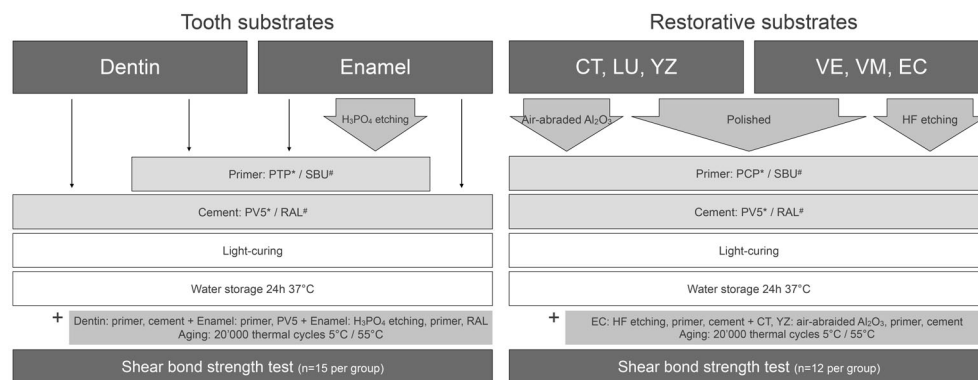


FIGURE 1 Progression of treatments within each experimental group for the shear bond strength test showing pretreatments of the substrates—to be read from top-to-bottom

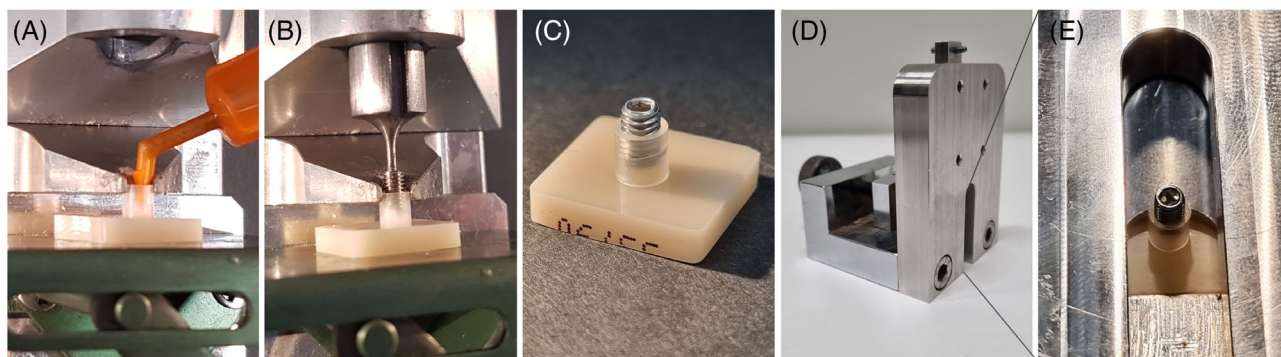


FIGURE 2 Test set-up: (A) Filling of cement into acrylic cylinder fixed on the pretreated substrate (B) Compressing cement with a screw followed by light-curing and subsequent water-storage (C) Specimen before shear bond strength test (D) Shear bond strength testing device (E) Shearing blade for load application in the universal testing machine

materials in the clinic and CT was selected to add a resin-based material to cover a wide selection of material types.

The shear bond strength test was performed using a crosshead speed of 1 mm/min in a universal testing machine (Z020, Zwick/Roell). The force at fracture was recorded (software testXpert III V1.51, Zwick/Roell) and shear bond strength was then calculated in MPa according to the debonded specimen cross-sectional area. Specimens displaying no bond were scored as 0 MPa.

Fractured surfaces were analyzed using light microscopy (VK-X1050, Keyence, Osaka Japan). The surfaces were then classified using the following modes: (1) cohesive failure within the substrate, (2) adhesive failure, and (3) cohesive failure within the cement.

2.3 | Statistics

Means and standard deviations of all groups were calculated. Normal distribution was validated using the Shapiro–Wilk test ($p < 0.05$). Compressive and diametral tensile strength values were compared using two-way ANOVAs to test the effect of cement type and curing mode, using Fisher's least significant difference (LSD) post-hoc tests ($\alpha = 0.05$). Two-way ANOVAs were performed on shear bond strength values of tooth and restorative substrates separately to test the effects of treatment and substrate ($\alpha = 0.05$) (without artificially aged specimens). Additional one-way ANOVAs followed by Fisher's LSD post-hoc tests were used to determine pair-wise differences within subgroups, including the aged samples.

3 | RESULTS

3.1 | Compressive and diametral tensile strength

The mean compressive strength for auto-polymerized PV5 was 312 ± 18 MPa, and 311 ± 19 MPa for light-cured PV5 specimens. For RUV, the mean compressive strength was 309 ± 35 MPa for auto-polymerized specimens and 328 ± 22 MPa for light-cured specimens.

There were no significant differences among these values ($p = 0.178$) as no effect was found for cement material ($p = 0.307$) or for polymerization mode ($p = 0.170$).

The mean diametral tensile strength value for PV5 (50 ± 5 MPa auto-polymerized, 50 ± 5 MPa light-cured) were significantly higher ($p < 0.001$) than for RUV (42 ± 4 MPa auto-polymerized, 42 ± 4 MPa light-cured). The curing mode did not have a significant effect for either PV5 ($p = 0.744$) or RUV ($p = 0.758$). Overall, the cement material significantly affected diametral tensile strength ($p < 0.001$) with higher values for PV5, while no effect was observed for the curing mode ($p = 0.638$).

3.2 | Shear bond strengths

Mean shear bond strength values and standard deviations with accompanying statistics are displayed in Table 2. For the tooth substrates, the highest values were achieved using PV5 on dentin and enamel when using the separate PTP primer (dentin: 18.0 ± 4.2 MPa, enamel: 18.0 ± 3.1 MPa), or following etch and prime (dentin: 16.9 ± 4.7 MPa, enamel: 20.9 ± 7.9 MPa) ($p < 0.001$). Aging of primed samples only affected shear bond strength to dentin for PV5 (12.2 ± 1.9 MPa) ($p < 0.001$), while values to enamel remained stable (19.7 ± 3.0 MPa) ($p = 0.973$). For RUV the highest values were recorded for dentin treated with SBU primer (18.2 ± 3.3 MPa) and for etched and primed enamel (21.2 ± 6.6 MPa) ($p < 0.001$). Aging of RUV on primed dentin (12.4 ± 4.6 MPa) as well as on etched and primed enamel (13.7 ± 3.8 MPa) resulted in significantly lower shear bond strength values ($p < 0.001$).

For the restorative material substrates, significantly higher shear bond strength values were achieved overall when substrates were micro-structured compared with the polished samples ($p < 0.001$). Bond strength to micro-structured EC (10.7 ± 3.3 MPa) ($p = 0.006$) was significantly higher when using PV5 compared with RUV, while for the other substrates stronger bonding was achieved using RUV (CT: 19.6 ± 6.0 MPa, $p < 0.001$; LU: 17.0 ± 3.5 MPa, $p < 0.001$; VE: 17.5 ± 2.7 MPa, $p < 0.001$; VM: 13.9 ± 1.3 MPa, $p = 0.002$; YZ:

TABLE 2 Shear bond strength values (mean and SD) in MPa. For tooth substrates $n = 15$, for restorative substrates $n = 12$ per group

Treatment	Cement/Primer	Tooth substrate					
		Dentin	Enamel	Dentin thermocycled	Enamel thermocycled		
In self-adhesive mode: no primer	PV5	0.0 ± 0.0 ^{A,a}	0.8 ± 1.3 ^{B,a}	-	-		
	RUV	10.8 ± 3.7 ^{A,b}	8.0 ± 3.6 ^{A,b}	-	-		
Primer used	PV5/PTP	18.0 ± 4.2 ^{A,c}	18.0 ± 3.1 ^{A,c}	12.2 ± 1.9 ^{B,a}	19.7 ± 3.0 ^{A,a}		
	RUV/SBU	18.2 ± 3.3 ^{A,c}	10.5 ± 1.8 ^{B,b}	12.4 ± 4.6 ^{B,a}	-		
Acid etched & Primer applied	PV5/PTP	16.9 ± 4.7 ^{A,c}	20.9 ± 7.0 ^{A,c}	-	-		
	RUV/SBU	12.5 ± 4.9 ^{A,b}	21.2 ± 6.6 ^{B,c}	-	13.7 ± 3.8 ^{A,b}		
		Restorative substrates					
		CT	LU	VE	VM	EC	YZ
Polished	PV5/PCP	0.0 ± 0.0 ^{A,a}	0.4 ± 0.8 ^{A,a}	3.9 ± 1.6 ^{B,a}	6.2 ± 1.3 ^{C,a}	3.6 ± 0.8 ^{B,a}	4.7 ± 1.2 ^{B,a,b}
	RUV/SBU	4.6 ± 1.8 ^{A,B,b}	5.9 ± 1.6 ^{A,b}	9.0 ± 2.3 ^{C,b}	3.7 ± 1.4 ^{B,b}	3.2 ± 1.2 ^{B,a}	12.4 ± 2.8 ^{D,c}
Micro-structured	PV5/PCP	0.0 ± 0.0 ^{A,a}	5.6 ± 3.6 ^{B,b}	13.6 ± 3.2 ^{C,c}	11.9 ± 1.8 ^{C,D,c}	10.7 ± 3.3 ^{D,b}	6.7 ± 1.6 ^{B,a}
	RUV/SBU	19.6 ± 6.0 ^{A,c}	17.0 ± 3.5 ^{A,B,c}	17.5 ± 2.7 ^{A,d}	13.9 ± 1.3 ^{B,d}	8.2 ± 1.1 ^{C,c}	16.8 ± 4.3 ^{A,B,d}
Micro-structured and thermocycled	PV5/PCP	-	-	-	-	2.1 ± 2.3 ^{A,a}	1.1 ± 1.5 ^{A,e}
	RUV/SBU	18.4 ± 6.8 ^{A,c}	-	-	-	7.1 ± 2.5 ^{B,c}	2.4 ± 3.1 ^{C,b,e}

Note: Values identified using similar letters (upper case—horizontal; lower case, vertical) are not significantly different. Statistical coding is limited to be within a type of substrate (tooth or restorative) only. Groups treated according to the recommendation of the manufacturer are highlighted. Cements: PV5: Panavia V5, RUV: RelyX Universal. Primers: PTP: Panavia V5 Tooth Primer, PCP: Clearfil Ceramic Primer Plus, SBU: Scotchbond Universal Adhesive Plus. Restorative substrates: CT: microfilled composite, LU: highly filled composite, VE: polymer-infiltrated ceramic, VM: feldspar ceramic, EC: lithium disilicate, YZ: zirconia.

TABLE 3 Incidences of failure modes occurring during shear bond strength testing. Number of failures: Type 1: cohesive within substrate; Type 2: adhesive; Type 3: cohesive within the cement. For tooth substrates $n = 15$, for restorative substrates $n = 12$ per group

Treatment	Cement/Primer	Tooth substrates					
		Dentin	Enamel	Dentin thermocycled	Enamel thermocycled		
In self-adhesive mode: no primer	PV5	0/15/0	0/15/0	-	-		
	RUV	4/10/1	0/15/0	-	-		
Primer used	PV5/PTP	8/5/2	3/12/0	2/12/1	4/11/0		
	RUV/SBU	10/2/3	0/15/0	5/8/2	-		
Acid etched & Primer applied	PV5/PTP	5/6/4	5/7/3	-	-		
	RUV/SBU	1/10/4	7/8/0	-	2/12/1		
		Restorative substrates					
		CT	LU	VE	VM	EC	YZ
Polished	PV5/PCP	0/12/0	0/12/0	0/12/0	10/2/0	0/12/0	0/9/3
	RUV/SBU	0/12/0	0/9/3	0/12/0	0/12/0	0/12/0	0/0/12
Micro-structured	PV5/PCP	0/12/0	0/12/0	12/0/0	12/0/0	0/1/11	0/9/3
	RUV/SBU	0/10/2	1/11/0	12/0/0	12/0/0	0/5/7	0/9/3
Micro-structured & thermocycled	PV5/PCP	-	-	-	-	0/12/0	0/12/0
	RUV/PCP	14/1/0	-	-	-	0/12/0	0/12/0

Note: Cements: PV5: Panavia V5, RUV: RelyX Universal. Primers: PTP: Panavia V5 Tooth Primer, PCP: Clearfil Ceramic Primer Plus, SBU: Scotchbond Universal Adhesive Plus. Restorative Substrates: CT: Microfilled composite, LU: highly filled composite, VE: polymer-infiltrated ceramic, VM: feldspar ceramic, EC: lithium disilicate, YZ: zirconia.

16.8 ± 4.3 MPa, $p < 0.001$) compared to PV5. Aging of micro-structured EC significantly reduced shear bond strength values for PV5 (2.1 ± 2.3 MPa, $p < 0.001$), while values for RUV remained stable

(7.1 ± 2.5 MPa, $p = 0.220$). For YZ, aging significantly reduced bond strength for both cements (PV5: 1.1 ± 1.5 MPa; RUV: 2.4 ± 3.1 MPa) ($p < 0.001$). A summary of the failure modes observed during the shear

bond strength tests for the different materials and treatments is presented in Table 3. In general, when bonding to tooth structures in self-adhesive mode, primarily adhesive failures (Type 2) occurred. Additional application of a primer increased cohesive failures within the substrate (Type 1), especially with dentin. Additional etching prior to primer application reduced cohesive failures (Type 1) again and more adhesive failures (Type 2) occurred on dentin, while for enamel cohesive failures within the enamel (Type 1) were increased. For restorative substrates that were polished, mainly adhesive failures (Type 1) occurred, except for YZ with cohesive failures within the cement (Type 3). When the restorative substrates were micro-structured (air-abraded or acid-etched), CT and LU displayed mainly adhesive failures (Type 2). For VE and VM cohesive failures (Type 1) within the restorative materials were predominant. For EC and YZ failures within the cement (Type 3) occurred, shifting toward adhesive failures (Type 2) after artificial aging.

4 | DISCUSSION

The first research hypothesis that the shear bond strength values to the tooth substrate and to selected restorative materials would be significantly higher when using the control adhesive resin cement (PV5) compared with the newly introduced self-adhesive cement (RUV) was rejected. Overall, the incorporation of several components into the universal adhesive RUV did not seem to limit, but rather improved the performance of the cement to restorative substrates. Bond strength to tooth structures remained even after artificial aging to dentin but was decreased more for RUV than for PV5 on enamel.

The second research hypothesis, that the compressive and diametral tensile strengths of the control adhesive resin cement (PV5) would be significantly higher compared with the newly introduced self-adhesive cement (RUV) was partially confirmed for the diametral tensile strength but rejected for compressive strength, where the values of both cements were not significantly different.

Resin composite cements are brittle materials, hence they are more susceptible to fail under tensile loading than with compressive stress.^{39,44} Compressive strength of a cement may be used to predict a restoration's resistance against masticatory forces.^{10–12,40} Materials demonstrating a low flexural strength such as silicate ceramics or composites, achieve a higher loading capacity on implants when cemented with adhesive cement having a compressive strength above 300 MPa, as measured for PV5 and RUV.^{11,12} Compared with the earlier 3 M cement products (RelyX Unicem 2 Automix and RelyX Ultimate), compressive strength of this latest product (RUV) was significantly increased.⁴¹ This result might be due to incorporation of ytterbium trifluoride filler in RUV replacing aluminum fluoride and silica present in the earlier versions.^{34,35} Also, the curing system of RUV demonstrates improved features, because the curing mode no longer affects the cement's strength compared with findings for the previous versions of this cement line: RelyX Unicem 2 Automix and RelyX Ultimate.⁴¹ To characterize the mechanical strength of resin composite cements, a three-point bending test is recommended by ISO standard

4049. However, the applied methods for compressive and diametral tensile strength testing with small specimen dimensions allow for an efficient and reproducible specimen production. Additionally, correlations of these methods to the flexural strength and Martens hardness have been thoroughly tested including aging procedures.^{41–44} Because the curing mode had no significant effect on compressive and diametral tensile strengths for either material, light-curing was performed to improve standardization of the specimen preparation.

Following the different bonding strategies, PV5 does not contain 2-hydroxyethyl methacrylate (HEMA) or MDP and consequently did not provide meaningful bonding to tooth substrates in the self-adhesive mode, which is in line with the indications given by the manufacturer. This specific cement is to always be used in combination with its tooth primer (PTP). On the other hand, RUV provided a certain adhesive bonding in its self-adhesive mode (without use of its universal bonding agent [SBU]) and may therefore be used for bonding to restorations having sufficient retention such as crowns, as recommended by the manufacturer. The cement RUV contains HEMA monomer that enhances the surface wetting, promotes resin penetration into demineralized dentin,^{23,24} and consequently enhances bonding to dentin.^{23,25,26} Additionally, phosphorylated dimethacrylates (mono-, di-, and tri-glycerol dimethacrylate ester of phosphoric acid) with a similar function as MDP are incorporated, which are able to form ionic bonds to calcium in hydroxyapatite, thus providing chemical bonding to enamel as well.²²

However, both cements exhibited their highest bond strength to dentin, when the respective primer was used. When the tooth primer (PTP) of the adhesive resin cement system PV5 was applied, as recommended by the manufacturer, high bond strengths to dentin and enamel were achieved demonstrating cohesive and adhesive failures. PTP is a self-etching primer having a pH of 2.0²⁷ that contains HEMA and MDP and is able to etch dentin and enamel without additional treatment with phosphoric acid. Because etching did not significantly improve the bond strength of PV5 to enamel, this cement might be used without any etching process. That observation is supported by earlier results, where the bond strength to etched and primed enamel was not significantly higher compared to the bond strength obtained after exclusive use of the respective primer without prior etching.²⁷

The application of the SBU primer for RUV is recommended for bonding to dentin, however for enamel, the present results clearly indicate that selective phosphoric acid etching is strongly advised prior to primer placement. SBU with RUV on dentin demonstrated the highest bond strength in all groups showing mainly cohesive failures Types 1 and 3. The bond strength to enamel was significantly lower, possibly due to the mild acidic composition of SBU (pH = 2.7 of precursor Scotchbond Universal Adhesive¹⁴) using acetic acid that is not sufficient to etch enamel. For PV5, phosphoric acid etching of dentin and enamel prior to priming had no additional effect on the shear bond strength. For RUV, the bond strength to dentin decreased significantly following etching, possibly due to a surface over-etching. However, the bond strength of RUV to enamel was almost doubled with the application of etching compared with no etching, and bond strength was still obtained after aging. Therefore, selective enamel

etching should always be performed with RUV, when the margin of the restoration is located in enamel, as for partial crowns or veneers. It is of great importance to note that the etching procedure must be performed precisely, as dentin may be accidentally etched, which would decrease bond strength in this specific area. It has to be kept in mind that only unaffected dentin was used in this investigation. In case of sclerotic dentin, a different approach might be beneficial which was not in scope of this investigation. The number of adhesive failures to the tooth substrates continuously decreased in favor of cohesive failures within the tooth substrates. For both cements the mean maximum shear bond strength was around 18 MPa for dentin and around 21 MPa for enamel, indicating the value of where the adhesive bond is stronger than the shear strength of the tooth tissues themselves.

To investigate the effect of artificial aging on shear bond strength to tooth substrates by thermal cycling, the most appropriate bonding procedures regarding bond strength and clinical handling were chosen. These were the use of primer without prior etching for PV5 and bonding to dentin with the use of primer as well as bonding to enamel with etching and priming for RUV. The approach for RUV corresponds to the selective etch technique. Bonding to dentin for both cements was significantly and in a similar extent affected by thermal cycling. The shear bond strength of RUV to enamel was significantly reduced by artificial aging. In contrast, the shear bond strength of PV5 to enamel was not affected by thermal cycling. It may be speculated that the bond to dentin, which is achieved via HEMA and MDP for both primers, relies mainly on dentin properties. However, for enamel other components of the respective primers seem to be crucial, hence the additional components of SBU, might have interfered with the bonding interface.

For bonding to restorative materials, it is essential to attain both a micro-mechanical interlocking and a chemical bond. To indirectly evaluate the potential for chemical bonding, a group of specimens were highly polished prior to bonding to minimize the effect of any type of micromechanical retention. Consequently, failures for polished substrates were mainly adhesive failures Type 2 and cohesive failures only occurred when the bond strength was higher than the strength of the restorative materials or cement itself.

Industrially produced CAD/CAM microfilled composites such as CT have a high degree of conversion and only limited numbers of free carbon-carbon double bonds available at the machined surface. Methyl methacrylate monomer can be applied,²⁰ but this has limited indication for intraoral use due to its high-allergic potential.²¹ No bonding of PV5 to CT was attained, even when the substrate surface was micro-structured. The primer PCP containing MDP and silane is probably rather hydrophilic and may not be compatible with a hydrophobic surface as demonstrated for CT.³⁶ For RUV, a slight bond was achieved on polished substrates which was significantly enhanced on micro-structured CT, but still associated with adhesive failures. Surprisingly, mainly Type 1 cohesive failures within the restorative substrate occurred when micro-structured CT was aged and the full bonding potential was maintained. This high-bonding capability to acrylate polymer of SBU might be attributed to acid groups of the "Vitrebond" copolymer, which may react with the acrylic groups of

CT. A second hypothesis might be that the penetration of primer components into CT and their polymerization is very effective.

The same effect as for CT was observed for the bond to LU for both cements. LU is a composite having a Bis-GMA/UDMA/TEGDMA resin matrix incorporating silica and zirconia fillers.^{12,46} The slight bond of PV5 to LU was possibly achieved due to silica particles via silane and to zirconia particles via MDP.

VE is composed of a porous continuous phase of etchable feldspar ceramic infiltrated with resin (TEGDMA/UDMA). As was observed for CT, PCP is not able to bond to a resin matrix, hence the bond to VE with PV5 must have been achieved through the silane via covalent Si—O—Si bonds to the feldspathic ceramic. The increase in bond strength and presence of cohesive failures confirmed that etching is a suitable procedure to micro-structure VE as has been previously observed.³ For RUV, bond strength to VE was slightly higher than for PV5 because SBU is able to bond to both the resin matrix via carbon double bonds and to silicate ceramic via silane.³

The bond to polished VM, a feldspar ceramic, was higher for PV5 than for RUV and mainly cohesive failures occurred, probably due to a strong chemical bond via silane. As has been reported for an earlier version of SBU, the silane within the universal adhesive displays reduced reactivity when mixed with too many components and consequently silanol polymerization and formation of low- and higher-order condensates occurs over that of silane interacting with the silica restorative substrate.^{3,14} Additional application of pure silane significantly improved the bonding performance of RelyX Ultimate and RelyX Unicem 2 Automix to VM with an earlier version of SBU.³ When VM was micro-structured in another study, bond strength values for RelyX Unicem 2 Automix were significantly higher than for PV5, possibly due to the filler content of SBU that interlocked well within the etched surface structure.³⁸

The same effect for polished EC was observed as for VM. When EC was etched, the topography was not as pronounced as for VM.³⁸ Consequently, PCP was able to better infiltrate into the micro-structure than was SBU with its higher viscosity. Because the diametral tensile strength of RUV is lower than that of PV5, more cohesive failures within the cement may have occurred with EC. However, after aging, values for RUV were more stable than for PV5. The silica filler of SBU might have been able to interlock mechanically with the etched EC, thus enhancing bond strength.

Bonding to zirconia is challenging, and MDP is currently considered as the most effective option.^{15,17} The phosphate group of MDP is able to engage in hydrogen bonding via P=O (oxo group) or non-deprotonated P—OH to zirconia or neighboring phosphate groups.¹⁷ Although it has been reported that the bond strength of the original MDP by Kuraray Noritake is higher than for less pure "copy-products,"¹⁸ bonding of RUV to YZ was significantly higher than for PV5 in this study. Due to the high-elastic modulus of zirconia, deformation occurred within the cement when the shear bond strength test was performed. Consequently, more cohesive failures occurred within PV5 and RUV in the polished and micro-structured YZ group. Aging resulted in a significant loss of bond strength to YZ for both cements.

The choice of cement system for a clinician is highly dependent on the type of preparation and restoration as well as individual preferences of the cementation system and cement viscosity. The primers of PV5 are of low viscosity, transparent, and evaporate quickly, while the universal adhesive SBU is yellow and more viscous due to its silica filler components, leaving a smeary layer on the surface. In general, when applied according to the manufacturer recommendations, both cements are indicated for clinical use based on their bonding performance.

When interpreting the present results, one must consider that stress distribution across the bonded interfaces may vary in a clinical situation. A clinically relevant cement layer thickness ranges from 30 to 300 μm ,^{8,9} while the cement layer in the present research was near 1500 μm . The tooth primer PTP of PV5 containing accelerators that contribute to the polymerization of PV5, was not used when testing restorative substrates, which is a further potential limitation of the present study. Previously, it has been shown that touch-curing with PTP significantly increased the polymerization degree of PV5 and consequently increased shear bond strength of PV5 to dentin and zirconia.³⁷ Therefore, bonding performance should be further evaluated in a controlled clinical setting.

Based on the data presented in the current work, it is suggested to use PV5 for restorations that rely on adhesive bonding with large dentin areas, such as partial crowns, because tooth pretreatment is faster (no etching required). For crowns with retentive features, RUV in the self-adhesive mode would be a better choice. For restorative materials primarily based on a resin matrix, which is not etchable with hydrofluoric acid (CT, LU), cementation with RUV is recommended, because no bonding is attained with PV5.

5 | CONCLUSIONS

Within the limitations of this current study, it was concluded that:

1. Compressive strengths of the adhesive resin composite cements PV5 and the newly introduced self-adhesive cement RUV were not significantly different, while the diametral tensile strength of PV5 was significantly higher.
2. Shear bond strength values to the tooth substrate and to selected restorative materials are not higher when using the adhesive resin composite cement PV5 compared with the newly introduced self-adhesive resin cement RUV.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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