

Repairability of Four CAD-CAM Materials

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Abstract

This study assessed the repairability among four CAD-CAM blocks repaired with different universal adhesives after aging. The CAD-CAM blocks evaluated were Polymer infiltrated ceramic network (Vita Enamic[®], VE), Zirconium-reinforced lithium silicate ceramic (Vita Suprinity[®], VS), Feldspathic ceramic (Vitablocs[®] Mark II, VM), and Lithium disilicate ceramic (IPS e.max[®] CAD, IE). Each original block was prepared into 5x5x5 mm dimensions to create 120 specimens. After being subjected to 10,000 cycles of thermocycling to simulate the oral environment conditions equivalent to one year of service, surface pretreatment was carried out by grinding and etching with a 9% hydrofluoric acid (Ultradent Porcelain Etch[®]). Subsequently, specimens were divided into three groups based on adhesive applied: Clearfil™ Tri-s bond Universal (CUB), Scotchbond Universal Plus[®] (SUP), and Monobond N[®] (MN, control group). Resin composite (Filtek™ Z350 XT, shade A3.5; 3M ESPE, USA) was then applied in a 2 mm thick-increment. All samples underwent another round of 10,000 cycles of thermocycling. A shear bond strength test (SBS) was performed, and the resulting data were analyzed by two-way ANOVA followed by Games-Howell or Least Significance Difference (LSD) post-hoc analysis ($P < 0.05$). Additionally, failure modes were examined under a stereomicroscope. Two-way ANOVA revealed significant impacts of both types of CAD-CAM blocks ($P < 0.001$), and universal adhesives ($P < 0.001$) on SBS values. Post-hoc analysis indicated that the SUP group exhibited improved repair SBS values compared with control and CUB groups ($P < 0.05$). VE + SUP group demonstrated the highest bond strength. The highest SBS was observed in the VE + CUB group ($P < 0.05$). Additionally, CAD-CAM material bonded with CUB showed significantly lower SBS compared with control groups in IE + CUB ($P < 0.05$). The lowest SBS was found in VM + MN ($P < 0.05$). In conclusion, universal adhesive containing 3-MPTES/APTES revealed superior repair bond strength across four CAD-CAM blocks. Particularly, hybrid ceramic and feldspathic ceramic exhibited favorable characteristics for repair with resin composite materials.

Keywords: CAD-CAM blocks, Ceramic surface treatment, Shear bond strength, Silane coupling agents, Universal adhesive

Received date: May 1, 2024 Revised date: May 14, 2024 Accepted date: Aug 2, 2024

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Introduction

Computer-aided design/computer-aided manufacturing blocks have emerged as favored materials for indirect restoration due to their numerous advantages, less time-consuming with superior esthetic outcomes, and reliable clinical performance.¹ Among these, machinable feldspathic porcelain, such as Vitablocs[®] Mark II by VITA Zahnfabrik, has been renowned for its higher mechanical properties compared to traditional porcelain. Notably, this block comprised fine-grain feldspathic porcelain with particles averaging 4 µm, enhancing microstructure homogeneity and fracture resistance.² Similarly, the popularity of glass-ceramics has increased due to their superior mechanical properties in comparison to leucite-glass ceramics.³ For instance, Lithium disilicate glass-ceramic (Li₂O₂SiO₂), such as IPS e.max[®] CAD (Ivoclar-Vivadent), was crafted by glass fusion and subsequent grinding into “blue blocks” for CAD-CAM.⁴

Another development in glass-ceramic materials involved the integration of polycrystalline ceramics to reinforce the vitreous matrix. Zirconium-reinforced lithium silicate (ZLS) materials, such as Vita Suprinity[®] (VITA Zahnfabrik), offered pleasing esthetics, good machinability and simple surface finishing due to their glass matrix content.⁵ Furthermore, polymer infiltrated ceramic network (PICN) materials have been introduced as a solution to the limitations of resin composite blocks, including their diminished durability and resistance to abrasive wear.⁴ Fabrication of PICN materials employed a two-step process. First, a porous pre-sintered ceramic network was treated with a silane coupling agent to enhance bonding. Followed by, a resin-based polymer infiltration, eliminating the need for a high-temperature post-firing step.⁶

Ceramic fracture or chipping has been a common failure in indirect restoration. Systematic reviews have identified ceramic onlay failure causes, including fracture, debonding, caries and other complications.⁷ Additionally, ceramic fractures were reported as the most prevalent failure mode across ceramic, composite, and hybrid ceramic.⁸ Several studies reported that ceramic chipping or cracking was considered a minor complication, defined as a cohesive

fracture not impairing function.^{9,10} Therefore, the ultimate replacement of ceramic restoration was not required since the dentist could either polish or repair it with resin composite.^{11,12}

Ensuring the longevity and reliability of resin composite-repaired ceramic restorations presented a significant challenge. It was crucial to ensure the bond durability between dental ceramic and resin composite, achieved through micro-mechanical retention methods such as hydrofluoric treatment and chemical retention via application silane.¹³ Studies suggested that a combination of hydrofluoric acid and silane provided optimal bond strength,¹⁴ with various silanes investigated, particularly in two-bottle systems demonstrating prolonged shelf life and enhanced reactivity.¹⁵ The development of universal adhesives represented a significant advancement in dental bonding technology. These adhesives achieved superior performance through a combination of a functional monomer, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), and a silane coupling agent, all conveniently pre-mixed in a single bottle. However, controversies surrounded the efficacy of this combination, with some studies indicating decreased bond strength with single step application compared to additional silane pretreatment.¹⁶

Despite the increasing utilization of resin composite for repairing indirect CAD-CAM restorations, there is a lack of definitive recommendations regarding the use of universal adhesives, and a universally accepted repair protocol has not yet been established. Thus, the present study evaluated the reparability among four CAD-CAM blocks repaired using different universal adhesives after aging.

Materials and methods

CAD-CAM blocks preparation

For specimen preparation, 120 specimens, each measuring 5x5x5 mm, were obtained by sectioning the original CAD-CAM blocks (initial dimensions: 12x14x18 mm) using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) (Fig. 1A). Then they underwent a thermocycling (KMITL, Bangkok, Thailand) protocol for

10,000 cycles using temperatures between 5°C and 55°C, with a dwell time of 30 seconds to simulate one year of oral service (Fig. 1B)¹⁷ The specimens were mounted in self-curing acrylic resin. This was followed by a 30-second polishing step using 320-grit silicon carbide abrasive papers under water irrigation. To ensure cleanliness, the specimens were then immersed in distilled water for ultrasonic cleaning for ten minutes. Table 2 details the chemical composition of the materials tested.

Surface pretreatment

The aged specimens underwent a surface pre-treatment protocol (n=120) involving etching with a 9% buffered hydrofluoric acid (Ultradent Porcelain Etch[®], Ultradent Products, Inc.) for various durations.

1. Vitablocs Mark II[®]: 9% HF for 60 seconds.
2. IPS e.max CAD[®]: 9% HF for 20 seconds.
3. Vita Enamic[®]: 9% HF for 60 seconds.
4. Vita Suprinity[®]: 9% HF for 20 seconds.

Afterward, all specimens were cleaned ultrasonically for ten minutes. The CAD-CAM surface-treated specimens were then categorized into three groups (n=40) based

on the adhesive applied: Clearfil[™] Tri-s Bond Universal (Kuraray Noritake Dental Inc), Scotchbond Universal Plus (3M, ESPE), and Monobond N (Ivoclar-Vivadent, Schaan/Liechtenstein).

Restorative procedure

To create a standardized bonding area, strips of one-sided ScotchBlue Painter's Tape (3M, Minnesota, USA) were cut into a 10x10 mm¹⁸, each with a central hole measuring 5 mm in diameter¹⁹. The tape itself was approximately 80 micrometers thick.¹⁸ Following this, the ceramic surface was covered with the tape. A micropipette was used to apply a drop of universal adhesive and silane coupling agent to each sample (10 microliters), with the solution then spread into a thin coat using a disposable applicator (Applicator tips, Dentsply DeTrey GmbH, Konstanz, Germany) (Fig. 1C). The LED light-curing unit (Demi[™] Plus, Kerr, USA) was subjected to calibration using an L.E.D. radiometer (DEMETRON, SDS Kerr, USA) after each day of use to ensure consistent light intensity. The chemical compositions of adhesive materials and details of application procedures were presented in Table 1.

Table 1 Manufacturers, compositions of tested materials

Material/ Manufacturer	Composition	Procedure following the manufacturer instructions
Monobond N [®] (Control) (Lot no. Z02XRS, Ivoclar-Vivadent,Schaan /Liechtenstein)	Ethanol, methacrylated phosphoric acid ester, sulphide methacrylate, 3-trimethoxysilylpropyl methacrylate	<ol style="list-style-type: none"> 1. Ceramic surfaces were rinsed and dried after pretreatment. 2. 10 µl of Monobond N[®] was applied with a disposable applicator. 3. The solution reacted with the specimen surface for 60 seconds. 4. Specimens were gently air-dried for 10 seconds until the absence of moving liquid droplets.
Scotchbond Universal Plus adhesive [®] (SUP) (Lot no.7910510, 3M ESPE Dental products)	10-MDP, HEMA, silane, dimethacrylate resins containing a BPA derivative-free, Vitrebond copolymer, filler, ethanol, water, initiators, dual-cure accelerator	<ol style="list-style-type: none"> 1. Ceramic surfaces were rinsed and dried after pretreatment. 2. 10 µl of Scotchbond Universal Plus adhesive[®] was applied (rubbing) to the entire ceramic surface and excess removed. 3. Specimen surfaces were gently air-dried until no liquid movement was observed with 2-bar pressure, from 10 mm distance. 4. The LED-light curing unit with 1,100 mW/cm² intensity was placed perpendicularly at a distance of 1 mm and adhesive was polymerized for 20 seconds.
Clearfil [™] TRI-S BOND Universal (CUB) (Lot no. 280057,	Bis-GMA, HEMA, MDP, CQ, colloidal silica, silane, ethanol, water,	<ol style="list-style-type: none"> 1. Ceramic surfaces were rinsed and dried after pretreatment. 2. 10 µl of Clearfil[™] TRI-S BOND Universal was applied (rubbing) and excess removed.

Table 1 Manufacturers, compositions of tested materials (cont.)

Material/ Manufacturer	Composition	Procedure following the manufacturer instructions
Kuraray Noritake Dental, Tokyo, Japan)	hydrophilic aliphatic dimetacrylate	3. Specimen surfaces were gently air-dried until no liquid movement was observed with 2-bar pressure, from 10 mm distance. 4. The LED-light curing unit with 1,100 mW/cm ² intensity was placed perpendicularly at a distance of 1 mm and adhesive was polymerized for 20 seconds.

Abbreviations: Bis-GMA, Bisphenol A-diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; BPA derivative-free, Bisphenol A derivative-free; CQ, Camphoroquinone

Following the bonding process, each specimen received a centrally placed hollow clear acrylic mold. The mold measured 5 mm in diameter and 4 mm in length. Then, a resin composite (Filtek™ Z350 XT, shade A3.5; 3M ESPE, USA) was applied to prepared specimens in 2 mm thick increments to fill the mold (Fig.1D). For each increment, an LED polymerization device (Demi™ Plus, Kerr, USA) delivered light from the top surface at 1,100 mW/cm² for 40 seconds. During the polymerization process, the tip of the light source was maintained in a perpendicular orientation, 1mm directly above the resin composite surface. After carefully removing the clear acrylic mold and tape, the specimen underwent an additional 40 seconds of irradiation to complete polymerization.

To simulate aging effects, all ceramic specimens underwent 10,000 thermocycles 5°C and 55°C with a 30

seconds dwell time (Fig. 1E). Subsequently, shear bond strength (SBS) was measured using a universal testing machine (EZ-S, Shimadzu, Japan). The SBS test involved applying a force at a constant speed of 0.5 mm/min using a knife-edge apparatus positioned between the CAD-CAM restorative material and the resin composite, with a separation distance of 1.0 mm (Fig. 1F). Shear stress on the specimens was progressively raised until they fractured. The force required to cause this failure was then documented (N). The SBS value was determined by dividing the highest SBS by the adhesive area (mm²). According to ISO29022:2013, a pre-test failure was recorded as 0 MPa. Any pretest failures, such as dislodgement of the composite button during removal of the button mold or excess composite, were noted accordingly.

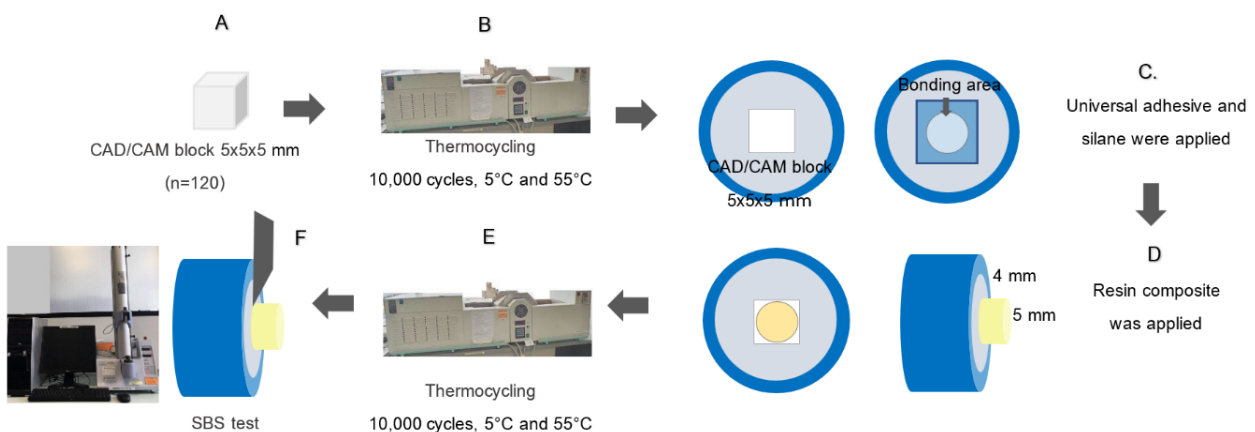


Figure 1 A. CAD-CAM blocks were sectioned into dimension of 5x5x5 mm to produce 120 specimens
B. Thermocycling was performed on all specimens
C. Bonding areas received a drop of adhesive (universal adhesives/silane coupling agent)
D. Following bonding procedure, resin composite was applied onto prepared specimens in 2 mm thick increments to fill up the mold
E. Prior to SBS testing, thermocycling was performed on all specimens
F. Subsequently, the SBS test was performed

Failure mode analysis

Mode of failure of the bonded resin-ceramic interface was randomly examined using a stereomicroscope (SZ 61, Olympus, Japan) at 2.5X magnification. Failures were categorized into three types: adhesive, cohesive, and mixed. Adhesive failure occurred at the interface between the adhesive and the ceramic restoration or between adhesive and composite or within the adhesive itself. Cohesive failure occurred within resin composite

or ceramic. Mixed failure exhibited both adhesive and cohesive failures in the fractured surface.

Scanning electron microscopy

Samples from different types of failure were randomly subjected to scanning electron microscopy (JEOL JSM-6610LV, Oxford X-Max 50) at 5000x magnification. The surface topographies were observed to confirm the result obtained from the mode of failure test.

Table 2 Compositions, manufacturers and components of ceramics and resin composite

Material	Compositions	Manufacturer	Lot number
Vita Enamic® (VE)	Ceramic part (86 wt%/75 vol%): SiO ₂ , Al ₂ O ₃ , Na ₂ O, K ₂ O, B ₂ O, ZrO ₂ , KaO Polymer part (14 wt%/25 vol%): UDMA, TEGDMA	VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany	79850
Vita Suprinity® (VS)	SiO ₂ , Li ₂ O, K ₂ O, P ₂ O ₅ , Al ₂ O ₃ , ZrO ₂ , CeO ₂ , pigments	VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany	96150
Vitablocs Mark II® (VM)	SiO ₂ , Al ₂ O ₃ , Na ₂ O, K ₂ O, CaO, TiO ₂	VITA Zahnfabrik H. Rauter GmbH & Co. KG, Germany	63240
IPS e.max CAD® (IE)	SiO ₂ , Li ₂ O, K ₂ O, MgO, Al ₂ O ₃ , P ₂ O ₅ and other oxides	Ivoclar-Vivadent, Schaan/Liechtenstein	Z024K2
Ultradent Porcelain Etch®	9% buffered hydrofluoric acid	Ultradent Dental Products, South Jordan, UT, USA	BM33P
Filtek™ Z350 XT Universal Restorative (A3.5)	UDMA, Bis-GMA, Bis-EMA, TEGDMA	3M ESPE Dental products, USA	NF31774

Abbreviations: UDMA, Urethane dimethacrylate; Bis-GMA; Bisphenol A-diglycidyl methacrylate; Bis-EMA, Bisphenol A-diglycidyl methacrylate ethoxylated; TEGDMA; Triethylene glycol dimethacrylate

Data analysis

The SPSS 20.0 for Mac was used to conduct a statistical analysis of the SBS for all groups. (SPSS Inc, Chicago, Illinois, USA). The findings indicated that the data followed a normal distribution, a two-way analysis of variance (ANOVA) was employed to analyze the effects of both material types and universal adhesives as main factors. Statistically significant difference was analyzed by one-way analysis of variance (ANOVA) followed by Games-Howell or Least Significance Difference (LSD) post-hoc analysis ($P < 0.05$) for multiple comparisons.

Results

Shear bond strength (SBS)

The results of SBS testing on repaired CAD-CAM blocks after surface treatments and universal adhesive application, including the mean SBS values, were shown in Table 3. Two-way ANOVA revealed that the types of CAD-CAM blocks ($F = 11.469, P < 0.001$), and universal adhesives ($F = 80.498, P < 0.001$) had a significant impact on SBS values (Table 1). In addition, the interaction between two factors were also significant (Table 3, $P < 0.001$).

Table 3 Influence of material (A) and universal adhesives (B) on Shear bond strength results according to Two-way ANOVA

Source	df	Sum of Squares Shear bond strength	Mean Square	F	P
Materials (A)	3	347.568	115.856	11.469	<.001
Universal adhesives (B)	2	1626.295	813.148	80.498	<.001
A x B	6	1069.012	178.169	17.638	<.001

Table 4 Mean shear bond strength (MPa) from four different types of CAD-CAM blocks and universal adhesives

Groups	Monobond N [®] Control group (MN)	CLEARFIL™ TRI-S BOND Universal (CUB)	Scotchbond Universal Plus [®] (SUP)
Vita Enamic [®] (VE)	7.335 ± (4.115) ^{aA}	13.400 ± (2.030) ^{aB}	19.551 ± (2.426) ^{aC}
Vita Suprinity [®] (VS)	10.302 ± (4.209) ^{acAC}	7.549 ± (1.966) ^{ba}	12.871 ± (3.825) ^{bbc}
Vitablocs Mark II [®] (VM)	0.187 ± (0.590) ^{ba}	8.501 ± (3.748) ^{bb}	18.049 ± (3.062) ^{bc}
IPS e.max CAD [®] (IE)	12.989 ± (4.180) ^{ca}	8.257 ± (2.660) ^{bb}	14.451 ± (3.120) ^{cbA}

Different small letters indicate significant differences within the same column. Different capital letters indicate significant differences within the same row. Statistically significant differences were analyzed by one-way ANOVA followed by Games-Howell or Least Significance Difference (LSD) post-hoc analysis ($P < 0.05$).

According to the post-hoc analysis, SUP improved in repair SBS values compared with the control and CUB groups (Table 4, $P < 0.05$). VE + SUP exhibited the highest bond strength values. There was no significant difference between VE + SUP and VM + SUP. However, the improvement obtained for IE + SUP and VS + SUP was not statistically significant compared with control groups ($P > 0.05$).

For CUB, the highest SBS was obtained from VE + CUB. On the other hand, there was no statistical difference among VS + CUB, VM + CUB and IE + CUB ($P > 0.05$). Additionally, CAD-CAM material bonded with CUB revealed significantly lower SBS compared with control groups in IE + CUB group. The lowest SBS was found in VM + MN.

Mode of failure

Table 5 summarized the distribution of failure modes and pretest failure scores observed in the SBS

testing of CAD-CAM materials. The predominant mode of failure was adhesive failure at the interface. In addition, the highest percent of cohesive failure was observed in VM + SUP. The mixed failure was observed in VE + MN, VE + CUB, VE + SUP, VM + CUB and VM + SUP groups. For the VE group, the highest percent of mixed failure was observed in VE + SUP, followed by VE + MN and VE + CUB, respectively. For VM, the highest percent of mixed failure was found in VM + CUB and VM + SUP. The pretest failures were observed in VM+MN group (Control).

Scanning electron microscopy

The SEM images (original magnification of 5000x) of CAD-CAM materials treated with various surface treatments, as shown in Figures 2-5, exhibited bonded surfaces after the SBS test to confirm mode of failure.

Table 5 Failure mode distribution

Material	Surface treatment	Adhesive	Cohesive	Mixed	Pretest failure
Vita Enamic [®] (VE)	Monobond N [®] (Control)	30% (3)	0	70% (7)	0
	Clearfil™ TRI-S BOND Universal (CUB)	50% (5)	0	50% (5)	0
	Scotchbond Universal Plus [®] (SUP)	0	0	100% (10)	0
Vita Suprinity [®] (VS)	Monobond N [®] (Control)	100% (10)	0	0	0
	Clearfil™ TRI-S BOND Universal (CUB)	100% (10)	0	0	0
	Scotchbond Universal Plus [®] (SUP)	100% (10)	0	0	0

Table 5 Failure mode distribution (cont.)

Material	Surface treatment	Adhesive	Cohesive	Mixed	Pretest failure
Vitablocs Mark II® (VM)	Monobond N® (Control)	10% (1)	0	0	90% (9)
	Clearfil™ TRI-S BOND Universal (CUB)	40% (4)	0	60% (6)	0
	Scotchbond Universal Plus® (SUP)	0	30% (3)	70% (7)	0
IPS e.max CAD® (IE)	Monobond N® (Control)	100% (10)	0	0	0
	Clearfil™ TRI-S BOND Universal (CUB)	100% (10)	0	0	0
	Scotchbond Universal Plus® (SUP)	100% (10)	0	0	0

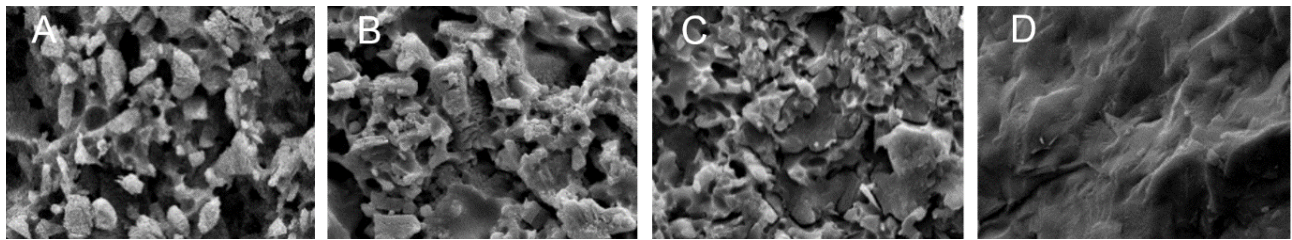


Figure 2 SEM of Vitablocs Mark II® (Original magnification x5000). 2A; After HF, the glassy phase partially dissolved. 2B; Adhesive failure in VM+MN. 2C; Mixed failure in VM+CUB. 2D; Cohesive failure within ceramic in VM+SUP

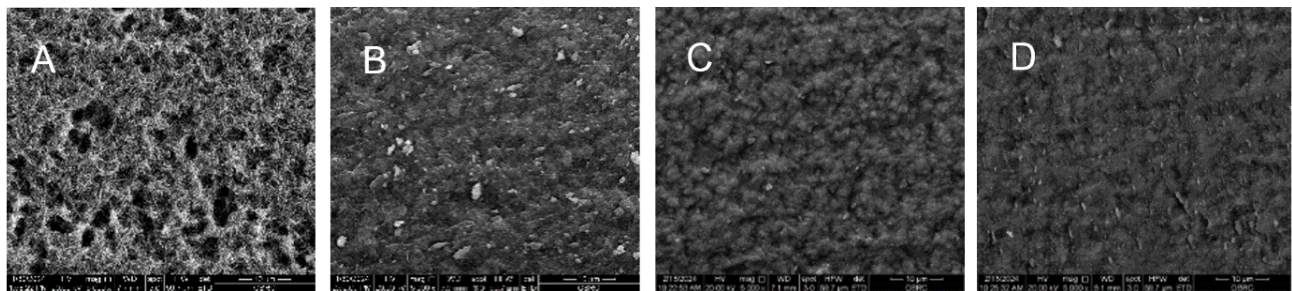


Figure 3 SEM of IPS e.max CAD® (Original magnification x5000). 3A; After HF, the glassy phase, partially dissolved phase, partially dissolved with predominantly crystalline phase. 3B; Adhesive failure in IE+MN. 3C; Adhesive failure in IE+CUB. 3D; Adhesive failure in IE+SUP

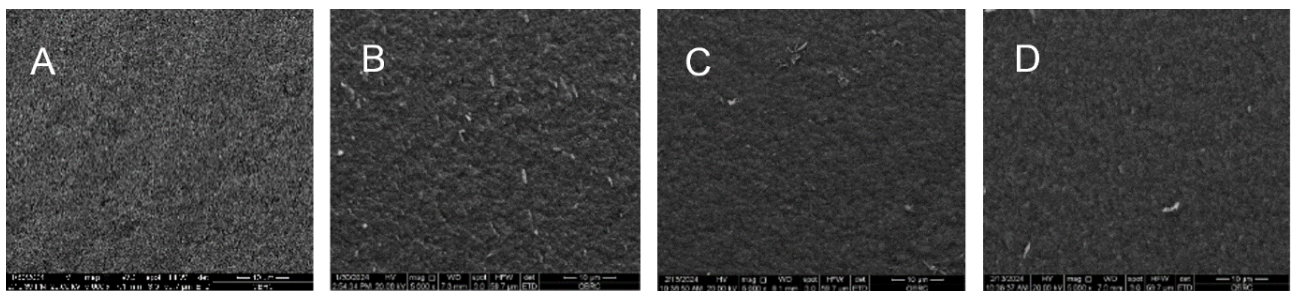


Figure 4 SEM of Vita Suprinity® (Original magnification x5000). 4A; After HF, the glassy phase partially dissolved with predominantly crystalline phase. 4B; Adhesive failure in VS+MN. 4C; Adhesive failure in VS+CUB. 4D; Adhesive failure in VS+SUP

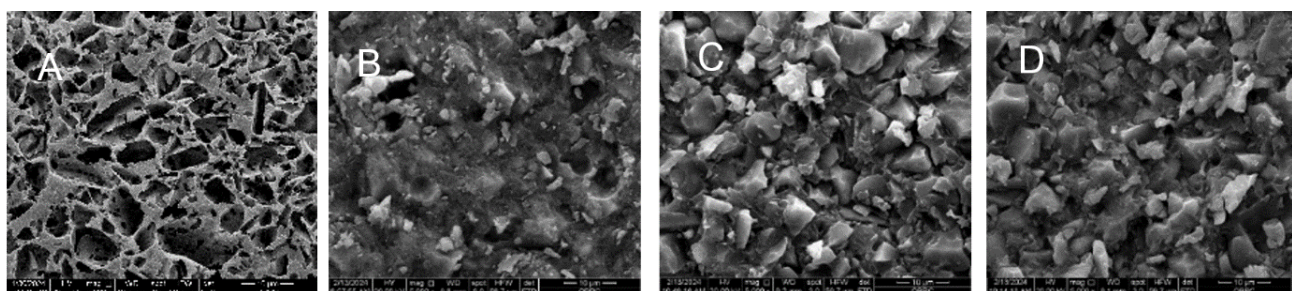


Figure 5 SEM of Vita Enamic® (Original magnification x5000). 5A; After HF, the glassy phase partially dissolved. 5B; Mixed failure in VE+MN. 5C; Mixed failure in VE+CUB. 5D; Mixed failure in VE+SUP

Discussion

This research conducted a comprehensive comparison of four CAD-CAM materials repaired using different universal adhesives. SUP demonstrated the highest SBS when applied with VE and VM. Moreover, it exhibited the highest SBS for VS and IE compared to CUB. These variations in performance could be attributed to differences in adhesive composition and microstructure of the materials.

The result of this study revealed that the types of CAD-CAM blocks significantly affected the SBS of the tested CAD-CAM blocks bonded to resin composite. The highest SBS for SUP and CUB was attained in the VE group. Polymer infiltrated ceramic network (PICN) material, essentially an interpenetrating phase composite, is formed by infusing a resin (14% by weight) into a ceramic network (86% by weight).²⁰ The previous study from Lima *et al.* indicated that unetched Vita Enamic[®] presented greater surface roughness compared to ZLS.²¹ The structure of Vita Enamic[®] exhibited porosity and possessed a composition similar to feldspathic ceramics.²² By selectively removing the glassy phase, acid etching modified the surface uniformity of the ceramic material, creating a rougher and potentially more uneven topography.²³ Additionally, combination of PICN within ceramic substrate contributed to the formation of a rougher surface. Another explanation is that monomers in universal adhesives enhance bond strength by interacting with reacted monomers in hybrid ceramics. Nevertheless, surface preparation remains essential to improve bonding with aged ceramics that do not contain unreacted monomers.²⁴

According to Straface *et al.*, the surface roughness of unetched Vitablocs Mark II[®] and Vita Enamic[®] measured 1.9 μm and 1.8 μm , respectively. However, Vita Suprinity[®] exhibited lower surface roughness. Another study demonstrated a relationship between etching time and the interaction area, indicating that longer etching times led to greater surface involvement.²⁵ Additionally, HF etching caused changes in surface topography, with higher concentrations resulting in more pronounced alterations and increased porosity.²⁶ A study by Azevedo confirmed that

feldspathic porcelain showed significant effects due to its higher glass content.²⁷ The use of 10% HF effectively dissolved the glassy matrix in glass-ceramics, creating a rough and retentive surface.²⁶ In their study, Straface *et al.* suggested extending hydrofluoric acid (HF) etching beyond 15 seconds, potentially up to 60 seconds, to guarantee comprehensive substrate etching and achieve complete dissolution of the glassy matrix.²²

The results of this research also indicated that the VM + MN group exhibited the lowest SBS. Similarly, VE + MN showed lower bond strength compared with IE + MN and VS + MN. Bonding between composite and ceramic depended on micromechanical and chemical bonds on ceramic surface.²⁸ Etching increased surface area, allowing uncured flowable resin to penetrate micropores and establish durable micromechanical interlocking.²⁸ To enhance chemical bonding between adhesive and resin composite, the ceramic surface underwent an etching process followed by treatment with a silane coupling agent.²⁹ Reactive silane groups bonded with hydroxyl groups on the ceramic surface, while the remaining non-hydrolyzable groups polymerized with the uncured resin composite.³⁰ However, this research aimed to investigate the reparability of CAD-CAM ceramic with conventional resin composite. Viscous universal composites were ineffective in penetrating micropores on ceramic surface after etching, resulting in uneven adaptation compared to resin composites with low filler content and liquid consistency.³¹ Thus, directly applying universal composite to silane-treated etched ceramic surfaces without adhesives could potentially affect SBS and lead to pretest failure in VM + MN groups. To address this issue, it is advisable to ensure the thorough application of the adhesive resin, allowing it to penetrate all etched ceramic surfaces and effectively interlock with them. This process anticipates the enhancement of the strength and durability of the repaired interface.

The results of this study also demonstrated that types of universal adhesives significantly affected the SBS of tested CAD-CAM blocks bonded to resin

composite. Compared to the MN and CUB groups, SUP showed significantly improved repair bond strength value. The presence of 3-MPTS in CUB did not affect SBS due to the dehydration-induced self-condensation of functional silanol in CUB led to instability of 3-MPTS molecules in acidic aqueous condition, consequently resulting in weaker bonding performance.^{32,33}

On the other hand, SUP contained a combination of functional monomer 3-(aminopropyl) triethoxysilane (APTES) and 3-methacryloxypropyltriethoxysilane (3-MPTES), enhancing the effectiveness of adhesive in priming glass ceramic surfaces.³⁴ The presence of 3-MPTES/APTES notably enhanced SBS between ceramic and composite compared to both the CUB and the control groups. After the glass-ceramic surface was etched, APTES molecules interacted with it by forming hydrogen bonds. These bonded APTES molecules then reacted with existing silanol groups, resulting in the creation of amino-silanol groups.³⁵ Moreover, the hydrolysis of Si-O-C₂H₅ group in 3-MPTES within SUP occurred at a slower rate in contrast to the Si-O-CH₃ group within 3-MPTS in the CUB. This delayed hydrolysis reduces the dehydration condensation of the silanol groups.³⁶

This study additionally demonstrated that CAD-CAM materials bonded with CUB exhibited lower SBS compared to control groups in VS + CUB and IE + CUB groups. There were no statistically significant differences in SBS among IE + CUB, VS + CUB and VM + CUB groups. Monobond N[®] comprised high proportions of organic solvent like ethanol.³⁷ When applied to an etched ceramic surface, Monobond N[®] formed a silane layer, which was an essential factor affecting the resin-ceramic bond strength. Unlike Monobond N[®], silane-containing universal adhesives consisted of notably complex compositions including 10-MDP, Bis-GMA, HEMA and others primarily designed to enhance bond strength. The presence of Bis-GMA resin slowed the condensation reaction between silanol groups of universal adhesive and ceramic by delaying evaporation of water.³⁸ As a result, the deposition of a silane on the ceramic surface seemed less feasible when using universal adhesives. This observation might explain the inferior priming efficacy

of universal adhesive compared to Monobond N[®] and Scotchbond Universal Plus[®]. This also explains the result that there were no statistically significant differences in SBS for IE + MN compared to IE + SUP and VS + MN compared to IE + SUP.

The study also revealed that VM + SUP had the highest incidence of cohesive failure, while VE + SUP showed the highest occurrence of mixed failure. This suggested that etching these ceramic types enhanced surface irregularities, thereby improving shear bond strength. Specifically, specimens repaired with SUP exhibited more cohesive and mixed failures compared to those repaired with CUB and MN. These results were consistent with the average bond strength findings, which showed that VE + SUP and VM + SUP achieved the highest bond strengths.

Thermocycling represented a prevalent approach in bond durability assessment, simulating oral cavity thermal changes induced by routine activities.¹⁷ This study observed no specimen failures during the thermal cycling process. Durability of the bond under thermal stress heavily relied on the number of cycles experienced, with temperature settings and dwell time also contributing.³⁹ Additionally, this study aimed to assess the reparability of aged CAD-CAM ceramic blocks. The specimens were subjected to 10,000 cycles, approximating one year of intraoral use before bonding procedure.¹⁷

This investigation focused on the influence of silane, specifically the type of silane used, within universal adhesives on ceramic repair. However, by focusing on only two silane-containing universal adhesives, the ability to assess the overall effectiveness of universal adhesives compared to traditional methods was limited. Including an additional group repaired with a conventional silane and adhesive would have allowed for a more comprehensive analysis of the bond strength achieved with different adhesive systems for ceramic repair. Another limitation was the absence of ceramic surface roughness assessment prior to the SBS test. Incorporating surface roughness tests would be advantageous for evaluating the impact of surface treatments before conducting the SBS evaluation.

For further studies, exploring different surface pretreatment methods, such as sandblasting and silica coating could be beneficial, as it may enhance bond strength compared to acid etching alone. This additional information would serve to validate the proper protocol for employing single-step universal adhesive systems for the repair of ceramic indirect restorations.

Conclusion

Within the limitations of this study, the universal adhesive containing 3-MPTES/APTES offered superior repair bond strength across four CAD-CAM blocks. Particularly, hybrid ceramic and feldspathic ceramic exhibit favorable characteristics when repaired with resin composite materials.

Declaration of conflicting interest

The authors declare no conflict of interest.

Acknowledgments

The authors gratefully acknowledge Assist. Prof. Dr. Soranun Chantarangsu for her valuable guidance on statistical analyses. Thanks is also extended to the staff of the Dental Material Research Centers and the Oral Biology Research Center of the Faculty of Dentistry, Chulalongkorn University.

References

- Liu PR. A panorama of dental CAD/CAM restorative systems. *Compend Contin Educ Dent* 2005;26(7):507-8,10,12
- Fasbinder DJ. Restorative Material Options for CAD/CAM Restorations. *Compend Contin Educ Dent* 2002;23(10):911-6,8,20
- Höland W, Schweiger M, Frank M, Rheinberger V. A comparison of the microstructure and properties of the IPS Empress®2 and the IPS Empress® glass-ceramics. *J Biomed Mater Res* 2000;53(4):297-303.
- Silva LHD, Lima E, Miranda RBP, Favero SS, Lohbauer U, Cesar PF. Dental ceramics: a review of new materials and processing methods. *Braz Oral Res* 2017;31(1):58.
- Sato TP, Anami LC, Melo RM, Valandro LF, Bottino MA. Effects of surface treatments on the bond strength between resin cement and a new zirconia-reinforced lithium silicate ceramic. *Oper Dent* 2016;41(3):284-92.
- Coldea A, Swain MV, Thiel N. Mechanical properties of polymer-infiltrated-ceramic-network materials. *Dent Mater* 2013;29(4):419-26.
- Abduo J, Sambrook RJ. Longevity of ceramic onlays: A systematic review. *J Esthet Restor Dent* 2018;30(3):193-215.
- Banh W, Hughes J, Sia A, Chien D, Kumar S, Figueredo C, et al. Longevity of Polymer-Infiltrated Ceramic Network and Zirconia-Reinforced Lithium Silicate Restorations: A Systematic Review and Meta-Analysis. *Materials (Basel)* 2021;14(17):5058.
- Fradeani M, D'Amelio M, Redemagni M, Corrado M. Five-year follow-up with Procera all-ceramic crowns. *Quintessence Int* 2005;36(2):105-13.
- Pallesen U, van Dijken JW. An 8-Year Evaluation of Sintered Ceramic and Glass Ceramic Inlays Processed by the Cerec CAD/CAM System. *Eur J of Oral Sci* 2000;108(3):239-46.
- Odman P, Andersson B. Procera AllCeram Crowns Followed for 5 to 10.5 Years: A Prospective Clinical Study. *Int J Prosthodont* 2001;14(6):504-9.
- Neis C, Albuquerque N, Albuquerque I, Gomes É, Souza-Filho C, Feitosa V, et al. Surface treatments for repair of feldspathic, leucite - and lithium disilicate-reinforced glass ceramics using composite resin. *Braz Dent J* 2015;26:152-5.
- Hooshmand T, van Noort R, Keshvad A. Bond Durability of the Resin-Bonded and Silane Treated Ceramic Surface. *Dent Mater J* 2002;18(2):179-88.
- Lacy AM, LaLuz J, Watanabe LG, Dellinges M. Effect of Porcelain Surface Treatment on the Bond to Composite. *J Prosthet Dent* 1988;60(3):288-91.
- Matinlinna JP, Lung CYK, Tsoi JKH. Silane Adhesion Mechanism in Dental Applications and Surface Treatments: A Review. *Dent Mater J* 2018;34(1):13-28.
- Yoshihara K, Nagaoka N, Sonoda A, Maruo Y, Makita Y, Okihara T, et al. Effectiveness and stability of silane coupling agent incorporated in 'universal' adhesives. *Dent Mater J* 2016;32(10):1218-25.
- Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent* 1999;27(2):89-99.
- Sriamporn T, Kraisintu P, See LP, Swasdison S, Klaisiri A, Thamrongananskul N. Effect of different neutralizing agents on feldspathic porcelain etched by hydrofluoric acid. *Eur J Dent* 2019;13(1):75-81.
- Visuttawattanakorn P, Rithy N, Suputtamonkol K, Kanchanasavita W. Shear bond strength of Zirconia to different adhesive resin cements. *M Dent J* 2015;35:127-36.
- Della Bona A, Corazza PH, Zhang Y. Characterization of a polymer-infiltrated ceramic-network material. *Dent Mater J* 2014;30(5):564-9.
- Albani R, Habib SR, AlQahtani A, AlHelal AA, Alrabiah M, Anwar S. The surface roughness of contemporary indirect CAD/CAM restorative materials that are glazed and chair-side-finished/polished. *Materials (Basel)* 2024;17(5):997.
- Straface A, Rupp L, Gintautė A, Fischer J, Zitzmann NU, Rohr N. HF etching of CAD/CAM materials: influence of HF concentration and etching time on shear bond strength. *Head Face Med* 2019;15(1):21.
- Queiroz-Lima G, Strazzi-Sahyon HB, Maluly-Proni AT, Fagundes TC, Briso ALF, Assunção WG, et al. Surface characterization

- of indirect restorative materials submitted to different etching protocols. *J Dent* 2022;127:104348.
24. Lucena-Martín C, González-López S, de Mondelo JMN-R. The effect of various surface treatments and bonding agents on the repaired strength of heat-treated composites. *J Prosthet Dent* 2001;86(5):481-8.
25. Ramakrishnaiah R, Alkheraif AA, Divakar DD, Matinlinna JP, Vallittu PK. The effect of hydrofluoric acid etching duration on the surface micromorphology, roughness, and wettability of dental ceramics. *Int J Mol Sci* 2016;17(6):822.
26. Murillo-Gómez F, Palma-Dibb RG, De Goes MF. Effect of acid etching on tridimensional microstructure of etchable CAD/CAM materials. *Dent Mater J* 2018;34(6):944-55.
27. Azevedo VLB, de Castro EF, Bonvent JJ, de Andrade OS, Nascimento FD, Giannini M, *et al.* Surface treatments on CAD/CAM glass-ceramics: Influence on roughness, topography, and bond strength. *J Esthet Restor Dent* 2021;33(5):739-49.
28. Nehir Özden A, Akaltan F, Can G. Effect of surface treatments of porcelain on the shear bond strength of applied dual-cured cement. *J Prosthet Dent* 1994;72(1):85-8.
29. Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. *Dent Mater J* 2012;28(5):467-77.
30. Matinlinna JP, Lassila LVJ, Ozcan M, Yli-urpo A, Vallittu PK. An introduction to silanes and their clinical applications in dentistry. *Int J Prosthodont* 2004;17(2):155-64.
31. Baroudi K, Rodrigues JC. Flowable Resin Composites: A Systematic Review and Clinical Considerations. *J Clin Diagn Res* 2015;9(6):Ze18-24.
32. Yao C, Yu J, Wang Y, Tang C, Huang C. Acidic pH weakens the bonding effectiveness of silane contained in universal adhesives. *Dent Mater J* 2018;34(5):809-18.
33. Yoshihara K, Nagaoka N, Sonoda A, Maruo Y, Makita Y, Okihara T, *et al.* Effectiveness and stability of silane coupling agent incorporated in 'universal' adhesives. *Dent Mater J* 2016;32(10):1218-25.
34. Yao C, Ahmed MH, De Grave L, Yoshihara K, Mercelis B, Okazaki Y, *et al.* Optimizing glass-ceramic bonding incorporating new silane technology in an experimental universal adhesive formulation. *Dent Mater J* 2021;37(5):894-904.
35. Park SJ, Jin JS, Lee JR. Influence of silane coupling agents on the surface energetics of glass fibers and mechanical interfacial properties of glass fiber-reinforced composites. *J Adhes Sci Technol* 2000;14(13):1677-89.
36. Xiong M, Zhang K, Chen Y. ATRP of 3-(triethoxysilyl)propyl methacrylate and preparation of "stable" gelable block copolymers. *Eur Polym J* 2008;44(11):3835-41.
37. Zakir M, Ashraf U, Tian T, Han A, Qiao W, Jin X, *et al.* The Role of Silane Coupling Agents and Universal Primers in Durable Adhesion to Dental Restorative Materials - a Review. *Curr Oral Health Rep* 2016;3(3):244-53.
38. Chen L, Shen H, Suh BI. Effect of incorporating BisGMA resin on the bonding properties of silane and zirconia primers. *J Prosthet Dent* 2013;110(5):402-7.
39. B. AFL, Colucci V, Palma-Dibb RG, Corona SAM. Assessment of *In Vitro* Methods Used to Promote Adhesive Interface Degradation: A Critical Review. *J Esthet Restor Dent* 2007;19(6):340-53; discussion 354.