

# A comparative study of microshear bond strength of hybrid CAD/CAM ceramic with universal bonding systems

Seelassaya Leelaponglit<sup>\*</sup>, Rebecca Kraivixien, and Sirivimol Srisawasdi

Department of operative dentistry, Faculty of dentistry, Chulalongkorn University, Bangkok, Thailand \*Corresponding author, E-mail: seelassaya@gmail.com

#### Abstract

The aim of this *in vitro* study was to evaluate the effect on microshear bond strength of various types of universal adhesive with dual-cure resin cement and different types of hybrid ceramic bonded to dentin. The flat buccal surfaces of 99 extracted human premolars were randomly allocated into nine groups. Three hybrid ceramics, Shofu block HC<sup>®</sup> (SHOFU, Japan), Vita Enamic<sup>®</sup> (VITA, Germany) and Cerasmart<sup>®</sup> (GC, USA), were prepared into an approximate size of 1 x 1 x 3 mm<sup>3</sup> slab and bonded to dentin using Scotchbond Universal<sup>®</sup> (3M ESPE, USA) with RelyX<sup>TM</sup>Ultimate<sup>®</sup> (3M ESPE, USA), Optibond XTR<sup>®</sup> (Kerr, USA) with Nexus3<sup>®</sup> (Kerr, USA) and Optibond FL<sup>®</sup> (Kerr, USA) with Nexus3<sup>®</sup> (Kerr, USA). After 10,000 cycles of thermocycling, all specimens were tested for microshear bond strength. Data were analyzed using a one-way ANOVA and Tukey's test ( $\alpha = 0.05$ ). The microshear bond strength in etch and rinse groups, Optibond FL<sup>®</sup> with Nexus3<sup>®</sup> in all hybrid ceramic tested. Within the limit of the study, there was a significant difference between the microshear bond strength when using various types of universal adhesive and resin cement systems with different types of hybrid ceramic. Vita Enamic<sup>®</sup> was recommended to cement with Optibond FL<sup>®</sup> and Nexus3<sup>®</sup>, whereas, Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup>Ultimate<sup>®</sup> was recommended to cement with Optibond FL<sup>®</sup> and Nexus3<sup>®</sup>, whereas, Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup>Ultimate<sup>®</sup> was recommended to cement with Optibond FL<sup>®</sup> and Nexus3<sup>®</sup>, whereas, Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup>Ultimate<sup>®</sup> was recommended to cement with Optibond FL<sup>®</sup> and Nexus3<sup>®</sup>, whereas, Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup>Ultimate<sup>®</sup> was recommended to cement with Optibond FL<sup>®</sup> and Nexus3<sup>®</sup>, whereas, Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup>Ultimate<sup>®</sup> was recommended to cement with Optibond FL<sup>®</sup> and Nexus3<sup>®</sup>, whereas, Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup>Ultimate<sup>®</sup>

**Keywords:** CAD-CAM resin ceramic, Hybrid ceramic, Microshear bond strength, Resin composite block, Resin matrix ceramic block, Universal bonding adhesive

#### 1. Introduction

Dental hybrid ceramics are material created with an attempt to mimic mechanical and optical properties of a natural tooth, possessing the properties of high strength and durability of dental ceramic with elastic modulus similar to dental hard structure (Spitznagel FA et al., 2014). These materials are easier to be milled and adjusted when compared to lithium disilicate group or polycrystalline ceramics, and easy to be repaired with composite resin (Gracis et al., 2015; He & Swain, 2011). Moreover, they have a higher degree of conversion, resulting in less monomer release, fewer voids, and improved mechanical properties compared to conventional direct resin composite material (Guth JF et al., 2012). The high pressure and high-temperature build-up in the process of manufacturing hybrid ceramic blocks resulted in improved flexural strength, elastic modulus, hardness, and density in comparison with traditional resin composite (Ruse ND & Sadoun MJ, 2014). However, hybrid ceramics have inferior optical properties (translucent and opalescence) than glass-ceramic; therefore, it was not popularly used in anterior teeth (Gunal| B & Ulusoy MM, 2017). These materials utilize computer-aided design and computer-aided manufacturing (CAD-CAM) technology to fabricate the indirect restoration. Currently, there are three types of hybrid ceramic (Gracis et al., 2015). 1) Resin block, such as Cerasmart<sup>®</sup> 2) Glass-ceramic in resin interpenetrating matrix, such as Vita Enamic<sup>®</sup> 3) Zirconia-silica ceramic in resin matrix, such as Shofu Block HC<sup>®</sup>.

Generally, indirect restoration requires the use of adhesives and resin cement for cementation. Adhesives can be mainly classified into two systems: etch and rinse system and self-etch system, according to the management of tooth surface (Pashley DH et al., 2011; Van Meerbeek B et al., 2011). Nowadays, most manufacturers aim to design adhesives for universal usage, which can be utilized in different modes (etch and rinse, self-etch or selective enamel etch) and bond to various substrates (Alex, 2015; Isolan CP et al., 2014; Moszner et al., 2005). A universal adhesive has similar nature as a one-step self-etch adhesive and contains special chemical compositions such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), silane. (C. Chen et al., 2015; Wagner A et al., 2014). Currently, 10-MDP has been used by many manufacturers, as a key component in universal bonding systems (Van Landuyt KL et al., 2007), which

[247]

**Proceedings of RSU International Research Conference (2020)** Published online: Copyright © 2016-2020 Rangsit University



demineralized and chemically interacted with hydroxyapatite creating MDP-Ca salt-forming nanolayered, resulting in durable bond to dentin (Van Landuyt KL et al., 2008; Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, et al., 2011). Etch and rinse adhesives increased the microscopic surface area available for resin monomer (Pashlev DH et al., 2011). These adhesives had statistically significant higher bond strength than some self-etch adhesives (Van Meerbeek B et al., 2011). The self-etch adhesive does not remove smear layer but interacts with smear layer (Giannini M et al., 2015; Van Meerbeek B et al., 2011; Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Torii Y, et al., 2011). Although the universal adhesive is designed to be used as a multi-mode system, some studies showed bond strengths of universal adhesives in self-etch mode are usually lower than those of two-and three-step etch and rinse adhesives (Sarr M et al., 2010). Currently, there are many commercially available universal adhesive products, in this study, we used Scotchbond Universal® (3M ESPE, USA). Long term bond strength and failure rate of Scotchbond Universal®, measured by using Fédération Dentaire Internationale (FDI) and United States Public Health Service (USPHS) criteria, showed no significant difference between post-18 month restoration and immediate restoration (Perdigao J et al., 2014). The success rate of the material was about 94-100%, while the failure rate was less than 10% (Perdigao J et al., 2014). Thus, using a universal bonding system to bond hybrid ceramic to tooth structure could be of interest. In this research, resin cement was used according to the manufacturer's recommendation, with the suggestion that adhesive and resin cement to be in the same system. We used a dual cure resin cement, which is suitable for the material when used as an indirect restoration.

Adhesion between the tooth structure and hybrid ceramic consists of two layers: the layer between the tooth structure with resin cement and the resin cement layer with hybrid ceramic. However, surface treatments, such as hydrofluoric acid etching or sandblasting with oxide particles combined with ceramic primers, are necessarily required for the material to increase adhesion efficiency (Awad et al., 2017; Murillo-Gomez et al., 2017). In this study, surface treatment was performed according to the manufacturer's recommendation. Shofu block HC<sup>®</sup> and Cerasmart<sup>®</sup> blocks that are resin-based were treated with aluminium oxide particle blast, followed by ceramic primer (Sturz et al., 2015). Vita Enamic<sup>®</sup> that has a composition like glass-ceramic was prepared with hydrofluoric acid. Then, the ceramic primer was applied (Stawarczyk et al., 2015).

Despite, several studies of the bond performance of hybrid ceramic bonded to dentin, there is limited information regarding the comparison of the bond performance of hybrid ceramics using various universal adhesives.

#### 2. Objectives

The objective of this study was to evaluate microshear bond strength ( $\mu$ SBS) of various universal adhesives and resin cement systems used to bond to different types of hybrid ceramic. The hypothesis was there was no significant difference between microshear bond strength when using various types of universal adhesive and resin cement systems with different types of hybrid ceramic.

#### 3. Materials and Methods

This study was approved by the Ethical Committee, Faculty of Dentistry, Chulalongkorn University, Thailand (Approval number: HREC-DCU 2018-050). All experimental procedures were performed by one investigator. Ninety-nine extracted human premolars with no caries or previous restoration, no cracks and free from debris/soft tissue were stored in a 0.1% thymol solution at  $4^{\circ}$ c. Dentin root was removed at 2-mm below the cementoenamel junction, using a water-cooled low-speed cutting machine, Isomet 1000 (Buehler, USA). Teeth were mounted in epoxy resin, with occlusal one-third buccal surface of dental crowns facing up and parallel to the horizontal plane. The epoxy resin was 5 mm below the buccal surface of the tooth and covered dentin root. The enamel of buccal surface was removed to expose a flat dentin area sized approximately 4x4 mm<sup>2</sup>. Dentin surfaces were then polished by a 600-grit wet silicon carbide paper at 100 RPM for 30 seconds. Specimens were randomly assigned into nine groups, according to bonding systems and hybrid ceramics (n=11).

Shofu block HC<sup>®</sup> shade A3 HT, Cerasmart<sup>®</sup> shade A3 HT, and Vita Enamic<sup>®</sup> shade 3M2 HT were cut into a 1x1x3 mm<sup>3</sup> block using a water-cooled low-speed cutting machine. Ceramic surfaces were

# [248]

**Proceedings of RSU International Research Conference (2020)** Published online: Copyright © 2016-2020 Rangsit University



polished by wet silicon carbide paper no. 120, 240, 400, and 600 grit, respectively. Surface treatment was performed according to the manufacturer's recommendations, as follows in Table 1.

All three types of hybrid ceramic blocks were bonded to dentin, using adhesives and resin cement in 3 systems according to the manufacturer's recommend; 1) Optibond  $FL^{\circledast}$  and Nexus  $3^{\circledast}$  2) Optibond  $XTR^{\circledast}$  and Nexus  $3^{\circledast}$  3) Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup> Ultimate<sup>®</sup>

Instructions for use are in Table 2.

Table 1 Surface treatment was performed according to the manufacturer's recommendations

Material	Surface treatment		
Shofu block HC®	1. Using 50-micron aluminium oxide particle blast, using a pressure of		
Cerasmart <sup>®</sup> blocks	0.2-0.3 bar, for 10 seconds.		
	<ol> <li>Treated with a ceramic primer Silane<sup>®</sup> (Ultradent, USA), and dried for 60 seconds.</li> </ol>		
Vita Enamic <sup>®</sup>	<ol> <li>Using a 9% hydrofluoric acid, Ultradent Porcelain Etch<sup>®</sup> (Ultradent, USA), for 90 seconds,</li> </ol>		
	2. Rinsed for 2 minutes, and dried.		
	3. Applied ceramic primer Silane <sup>®</sup> and air-dried for 60 seconds.		

After adhesive and resin cement application, the one-newton load was applied, from Durameter, ASTM D2240 Type A (PTC Instrument, USA), when pressing the material onto the tooth. Then, cement excess was removed and light-cured for 40 seconds on each side using LED Curing light Demi <sup>TM</sup> Plus (Kerr, USA), with an intensity more than  $1,000/\text{cm}^2$ . Light intensity was checked every 10 times of use, using a radiometer Light Intensity Meter 100 Optilux (SDS/Kerr, USA). Subsequently, the artificial aging process by thermocycling was performed, via cold and hot water temperature at 5°C and 55°C for 10,000 cycles with a dwell time of 30 seconds for each bath. After thermocycling process, microshear bond strength was tested by a universal testing machine EZ-S (SHIMADZU, Japan) using a force of 5 newtons with a cross-head speed of 0.5 millimeters per minute until the material was broken and the value recorded in megapascals (MPa).

After the specimen fractured, failure characteristics were examined with the SZ 61 stereomicroscope (Olympus, Japan) at 40X magnification. Then, failure characteristics were recorded according to fracture types: 1) adhesive failure mode which the fracture was found at the interface of adhesive and dentin or hybrid ceramics, 2) cohesive failure mode which the fracture was found within the substrate and 3) mix failure mode which the fracture was found in an adhesive layer with dentin and hybrid ceramic. Representatives of 2 samples per group were randomly chosen to examine the failure characteristics by a scanning electron microscope QUANTA<sup>TM</sup>250 (FEI company, USA), at a 3,500X magnification.

## Statistical analysis

Statistical data were analyzed by an SPSS Version 22. Data distribution was determined by a Kruskal Wallis test. Microshear bond strength values were compared between types of adhesive and resin cement, with different types of hybrid ceramic. A one-way ANOVA was used to compare the significant difference in microshear bond strength between types of universal adhesive and resin cement systems in each type of hybrid ceramics. Difference between mean values was evaluated using a Tukey's post hoc multiple comparison test at the 0.05 level of significance.



Adhesive and resin cement	Chemical composition	Instructions for use according to the manufacturer's recommendations
Optibond FL <sup>®</sup>	Etchant: 37.5% phosphoric acid Primer: HEMA, GPDM, PAMM, CQ, ethanol, water (3093079); adhesive: TEGDMA, UDMA, Bis-GMA, HEMA, GPDM, filler, CQ (3096500)	<ul><li>Etch dentin with 37.5% phosphoric acid for 15 seconds.</li><li>Rinse for 10 seconds and dry for 5 seconds.</li><li>Apply primer with light brushing motion for 15 seconds, air-dry for 5 seconds.</li><li>Apply adhesive with light brushing motion for 15 seconds, air-dry for 5 seconds.</li></ul>
Optibond XTR <sup>®</sup>	Primer: GPDM, hydrophilic mono- and di- functional methacrylate, water, acetone, ethanol, CQ Adhesive: hydrophobic, structural and cross-linking monomers, ethanol, CQ, barium glass filler, nano-silica filler	Apply primer to dentin surface, scrub with a brushing motion for 15 seconds and with the air thin for 10 seconds with medium air pressure. Apply adhesive to dentin surface with light brushing motion for 10 seconds and with the air thin for 5 seconds.
Nexus 3 <sup>®</sup>	Base: methacrylate ester monomers, HEMA, 2-PTU (pyridylthiourea) Catalyst: methacrylate ester monomers, HEMA, CHPO (cumene hydroperoxide), TiO <sub>2</sub> pigments	Using auto mix cement tip to apply to the material. Light cure for 40 seconds.
Scotchbond Universal <sup>®</sup>	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer filler, ethanol, water, initiators, silane	<ul><li>Etch dentin with 37% phosphoric acid for 15 seconds.</li><li>Rinsed for 10 seconds, and dried for 5 seconds.</li><li>Adhesive applied to the ceramics for 20 seconds.</li><li>Adhesive gently air-dried for approximately 5 seconds</li></ul>
RelyX <sup>™</sup> Ultimate <sup>®</sup>	10-Methacryloxydecyl dihydrogen phosphate (MDP), dimethacrylate resins, HEMA, Vitrebond <sup>TM</sup> copolymer filler, ethanol, water, initiators, silane	Using auto mix cement tip to apply to the material. Light cure for 20 seconds.

## Table 2 Adhesive and resin cement used in this study

# 4. Results and Discussion

#### Results

The microshear bond strength values were normally distributed. Means and standard deviations (SD) of three hybrid ceramics bonded with three adhesives and resin cement systems were shown in table 3.

Table 3 Mean $\pm$ SD of microshear bond strength	values in each group of three hybrid ceramics bonded with three
adhesives and resin cement systems (MPa)	

	Optibond FL <sup>®</sup> + Nexus3 <sup>®</sup>	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup>	Scotchbond Universal <sup>®</sup> + RelyX <sup>TM</sup> ultimate <sup>®</sup>
Shofu block HC®	$25.20 \pm 7.93$ <sup>a,A</sup>	$13.05 \pm 3.25$ <sup>b,A</sup>	$40.62 \pm 10.50$ <sup>c</sup>
Vita Enamic®	$53.07 \pm 11.43$ <sup>a,B</sup>	$24.55 \pm 7.21$ <sup>b,B</sup>	$36.68 \pm 7.14$ <sup>c,A</sup>
Cerasmart®	$39.77 \pm 11.11$ <sup>a,C</sup>	$22.55 \pm 3.41$ <sup>b,B</sup>	$48.68 \pm 10.52 \ ^{a,B}$

Remarks:

No statistical significance among groups with the same uppercase letter in each column (p > 0.05) No statistical significance among groups with the same lowercase letter in each row (p > 0.05)

# [250]

**Proceedings of RSU International Research Conference (2020)** Published online: Copyright © 2016-2020 Rangsit University



There was a statistically significant difference of microshear bond strength between types of hybrid ceramic and types of dental adhesives paired with resin cement, as shown in Table 4. Therefore, the null hypothesis was rejected.

It was shown that Vita Enamic<sup>®</sup> bonded with Optibond FL<sup>®</sup> and Nexus3<sup>®</sup> showed the highest microshear bond strength. The result revealed a statistically significant difference compared to other groups of the bonding system.

Table 4 One-way ANOVA and Tukey's post hoc multiple comparison test used to statistically analyze µSE	S between
groups	

Materials	Adhesive and resin cement systems		Sig. (Post Hoc)	F	Sig.
	Optibond FL <sup>®</sup> + Nexus3 <sup>®</sup> - Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup> -	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup>	.003	- 34.307	
		Scotchbond Universal <sup>®</sup> + RelyX <sup>TM</sup> ultimate <sup>®</sup>	.000		
		Optibond FL <sup>®</sup> + Nexus3 <sup>®</sup>	.003		
Shofu block HC <sup>®</sup>		Scotchbond Universal <sup>®</sup> + RelyX <sup>TM</sup> ultimate <sup>®</sup>	.000		.000
	Scotchbond	Optibond FL <sup>®</sup> + Nexus3 <sup>®</sup>	.000	-	
	Universal <sup>®</sup> + - RelyX <sup>TM</sup> ultimate <sup>®</sup>	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup>	.000	-	
	Optibond FL <sup>®</sup> +	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup>	.000	- 28.927 .(	
	Nexus3 <sup>®</sup> - Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup> -	Scotchbond Universal <sup>®</sup> + RelyX <sup>TM</sup> ultimate <sup>®</sup>	.000		.000
		Optibond FL <sup>®</sup> + Nexus3 <sup>®</sup>	.000		
Vita Enamic®		Scotchbond Universal <sup>®</sup> + RelyX <sup>TM</sup> ultimate <sup>®</sup>	.008		
	Scotchbond	Optibond FL <sup>®</sup> + Nexus3 <sup>®</sup>	.000		
	Universal <sup>®</sup> + - RelyX <sup>TM</sup> ultimate <sup>®</sup>	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup>	.008	-	
Cerasmart®	Optibond FL <sup>®</sup> +	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup>	.000		
	Nexus3 <sup>®</sup> –	Scotchbond Universal <sup>®</sup> + RelyX <sup>TM</sup> ultimate <sup>®</sup>	.070	_	
	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup> -	Optibond FL <sup>®</sup> + Nexus3 <sup>®</sup>	.000	23.676	.000
		Scotchbond Universal <sup>®</sup> + RelyX <sup>TM</sup> ultimate <sup>®</sup>	.000		
	Scotchbond	Optibond FL®+ Nexus3®	.070		
	Universal <sup>®</sup> + - RelyX <sup>TM</sup> ultimate <sup>®</sup>	Optibond XTR <sup>®</sup> + Nexus3 <sup>®</sup>	.000	-	

Among samples in Cerasmart<sup>®</sup> group, the highest microshear bond strength was found in the sample using Scotchbond Universal<sup>®</sup> combined with RelyX<sup>TM</sup> ultimate<sup>®</sup>, which was not significantly different when compared them to Cerasmart<sup>®</sup> with Optibond FL<sup>®</sup> combined with Nexus3<sup>®</sup>. Shofu block HC<sup>®</sup> bonded to dentin with Scotchbond Universal<sup>®</sup> combined with RelyX<sup>TM</sup>ultimate<sup>®</sup> had a statistically significant highest microshear bond strength compared to other groups of the bonding system. All three ceramic materials using Optibond XTR<sup>®</sup> and Nexus3<sup>®</sup> showed the lowest microshear bond strength values. Failure types were classified by groups, as shown in Figure 1.

# [251]



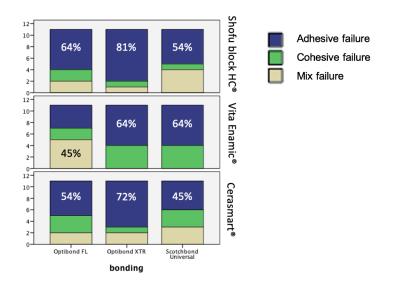
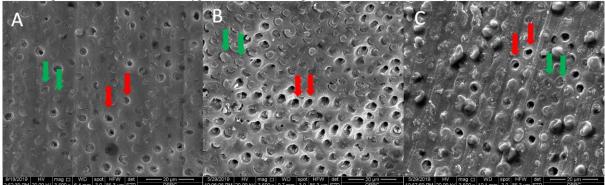
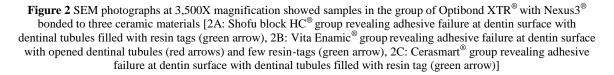


Figure 1 Number of failure types following microshear bond strength test

Two samples in each group were randomly selected to be examined by a scanning electron microscope and to confirm failure types at a 3,500X magnification, as shown in Figure 2.





# Discussion

This study compared the effect of various luting systems on microshear bond strength between hybrid ceramic and dentin. Three hybrid ceramic materials available in Thailand, Shofu block  $HC^{\text{(B)}}$ , Vita enamic<sup>®</sup>, and Cerasmart<sup>®</sup>, were used. The universal adhesives were used with dual-cure resin cement from the same manufacturer, including Scotchbond Universal<sup>®</sup> with RelyX<sup>TM</sup>ultimate<sup>®</sup> resin cement, and Optibond XTR<sup>®</sup>, as the universal adhesive of a two-step self-etch system, with Nexus3<sup>®</sup>. The control group was Optibond FL<sup>®</sup> with Nexus3<sup>®</sup>, in which Optibond FL<sup>®</sup> or three-step etch and rinse adhesive was considered as the standard control group, due to long track records. All specimens underwent an artificial aging process using 10,000 cycles of thermocycling, equivalent to 1 year of use in the oral cavity (Blumer et al., 2015).

# [252]

**Proceedings of RSU International Research Conference (2020)** Published online: Copyright © 2016-2020 Rangsit University



Considering adhesion of all three hybrid ceramic materials, Scotchbond Universal® and Optibond FL<sup>®</sup> with resin cement, used in the etching and rinse mode, showed statistically significant higher bond strength than Optibond XTR<sup>®</sup> with Nexus3<sup>®</sup>. The results of this study found higher bond strength of etching and rinse adhesive system compared to the self-etch adhesive system. It may be explained by the effect of phosphoric acid which got rid of debris and smear layer on the dentinal surface and in dentinal tubules dissolving hydroxyapatite crystals and exposed collagen network. Therefore, the use of phosphoric acid etching before an adhesive application may provide deeper hybridization to dentin resulting in higher density and possibly longer resin tags of hybrid layer, consequently higher bond strength was achieved (Ceballos et al., 2003) (Ikeda et al., 2008; Wagner A et al., 2014). The entire adhesive application process provided a micromechanical resin-dentine bond that could resist various insults (Van Meerbeek B et al., 2005). On the other hand, Optibond XTR<sup>®</sup>, mild self-etch adhesive (primer pH of 2.4), presented the lowest microshear bond strength when used to bond the hybrid ceramic. It is possible that the hydrophilicity of the self-etch adhesive system made hybrid layer more prone to hydrolytic degradation of polymer ester bonds (De Munck et al., 2010). These results were supported by the predominant adhesive failure mode (SEM) of Optibond XTR<sup>®</sup> and Nexus3<sup>®</sup>, as shown in figure 1 and figure 2, of which the exposed dentin surface was revealed with exposed dentinal tubule.

Based on the composition, hybrid materials in this study may be subdivided into materials that are based on resins (CAD/CAM resin composite: Shofu block HC and Cerasmart<sup>®</sup>) or ceramics (hybrid ceramic: Vita Enamic<sup>®</sup>). Scotchbond Universal<sup>®</sup> adhesive presented the highest bond strength when used to bond to Shofu block HC<sup>®</sup> and Cerasmart<sup>®</sup>. These results were possibly due to 10-MDP and PAC functional monomers of Scotchbond Universal<sup>®</sup> adhesive, which interacted with calcium in hydroxyapatites. The 10-MDP monomer has been identified to have high chemical bonding potential to chemically interact with hydroxyapatites establishing MDP-Ca water-insoluble crystals in the form of nanolayer (Wagner A et al., 2014), both on enamel and dentin, (Hoshika S et al., 2018; Iwai H & N., 2012; Oliveira BMB et al., 2017; Tian F et al., 2016; Wang et al., 2017; Yoshida Y et al., 2012; Yoshida Y. et al., 2004; Yoshihara K et al., 2015; Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, et al., 2011). These stable nanolayer structures may provide strong and durable adhesive bond strength. As the study of Darbanne et al. showed that the use of 10-MDP containing adhesive system bonded to dentin had higher shear bond strength with statistical significance than the use non-10-MDP containing adhesive (Derbanne MA et al., 2014). Moreover, Muñoz et al. revealed that the combination of PAC and 10-MDP resulted in higher bond strength than adhesive containing only 10-MDP (Munoz MA et al., 2015).

Optibond FL<sup>®</sup> presented the highest bond strength when used to bond to Vita Enamic<sup>®</sup>. This results possibly occurred as Vita Enamic® was composed of 86 % by weight feldspar ceramic. The matrix components were infiltrated UDMA and TEGDMA, with less than 1 % by weight of 200-400 nm size zirconia filler. In the production process, this material was considered to be the only true hybrid type of ceramic (Da Silva LH et al., 2017). Noda et al., showed that Scotchbond Universal®, bonded to silicacontaining ceramics, such as lithium disilicate and feldspar, was not good due to the composition of 3methacrylxypropyl trimethoxysilane (y-MPTS) that decomposed when mixing with elements with HEMA and water, leading to the reduction of bond strength and accelerated (Noda et al., 2017) the process of water absorption into the adhesive layer during the artificial aging process (Tsujimoto et al., 2017). The universal adhesive, Scotchbond Universal®, contained silane which could be unstable with a short lifespan and easy disintegration (Yao C et al., 2018). This study applied silane separately before adhesion. The process of thermocycling may result in the breakdown of siloxane bond from absorption of water, and consequently lower bond strength (Cardenas et al., 2017; Matinlinna JP & Lassila LV, 2010). In contrast, application of Scotchbond Universal<sup>®</sup> did not decrease the bond strength of resin composite bonded to dentin (Isolan CP et al., 2014). Therefore, when using Scotchbond Universal® paired with Rely X ultimate® to cement Vita Enamic<sup>®</sup>, lower microshear bond strength was achieved compared to other adhesives.

In this study, the ceramic surface treatments were done as recommended by the manufacturers. Shofu block HC<sup>®</sup> and Cerasmart<sup>®</sup>, of resin-like composition, were sandblasted with aluminum oxide particles to create microroughness (Blatz et al., 2003; Soares CJ et al., 2005; Terry & Blatz, 2010). Then,

[253]



the ceramic primer or silane was used. Vita Enamic<sup>®</sup>, of glass-like composition, was treated with 9 % hydrofluoric acid to create surface roughness (Terry & Blatz, 2010) for the disclosure of crystal structure creating a large number of small pores and increasing size of surface area (Tian et al., 2014), to facilitate penetration of resin cement into the ceramic surface (Blatz et al., 2003). Subsequently, the ceramic primer was used to enhance chemical adhesion (Hori et al., 2008). This ceramic primer was composed of molecules with a bi-functional group to stimulate siloxane bonding between silica crystals on the ceramic glass surface and methacrylate groups of resin cement (Aida et al., 1995; Hori et al., 2008). The ceramic surface treatment promoted better adhesion between tooth structure and ceramic by increasing surface energy (Strasser et al., 2018), wettability, and bond strength value (Kalavacharla V et al., 2001). However, different surface treatments, according to types of material, was considered as a crucial factor for types of ceramic. Therefore, further studies into surface preparation using other mechanisms, other chemical agents or surface treatment technique may be recommended. The surface treatment in this research was specified following the manufacturer's recommendations. Also, different surface treatments may result in different bonding performances.

Based on the results of the present study, the mode of the adhesive system and the composition of hybrid materials seem to influence bond strengths. This study was a laboratory test using only some universal adhesives available in the market with a restriction on sample preparation; thus, the outcomes could not truly be compared with clinical performance of all three types of hybrid ceramic. Hence, further studies on the long-term clinical applications, surface treatment technique and the use of other universal adhesives and resin cement are recommended.

### 5. Conclusion

When attaching the hybrid ceramic material to the dentin, types of adhesives and types of hybrid ceramic had impacts on microshear bond strength significantly. When using Vita Enamic<sup>®</sup>, it is recommended to cement with Optibond  $FL^{\$}$  and Nexus3<sup>®</sup>, whereas, Shofu block HC<sup>®</sup> and Cerasmart<sup>®</sup> are suggested to bond with Scotchbond Universal<sup>®</sup> and RelyX<sup>TM</sup>Ultimate<sup>®</sup>.

#### 6. Acknowledgements

I would like to express my sincere thanks to Assist. Prof. Dr. Soranun Chantarangsu for the statistical consultation, and to the Dental Biomaterials Science Center, Chulalongkorn university for their research assistance.

# 7. References

- Aida, M., Hayakawa, T., & Mizukawa, K. (1995). Adhesion of composite to porcelain with various surface conditions. J Prosthet Dent, 73(5), 464-470.
- Alex, G. (2015). Universal adhesives: the next evolution in adhesive dentistry? Compend Contin Educ Dent, 36(1), 15-26.
- Awad, M. M., Alqahtani, H., Al-Mudahi, A., Murayshed, M. S., Alrahlah, A., & Bhandi, S. H. (2017). Adhesive bonding to computer-aided design/computer-aided manufacturing esthetic dental materials: an overview. J Contemp Dent Pract, 18(7), 622-626.
- Blatz, M. B., Sadan, A., & Kern, M. (2003). Resin-ceramic bonding: a review of the literature. J Prosthet Dent, 89(3), 268-274. doi:10.1067/mpr.2003.50
- Blumer, L., Schmidli, F., Weiger, R., & Fischer, J. (2015). A systematic approach to standardize artificial aging of resin composite cements. *Dent Mater*, *31*(7), 855-863. doi:10.1016/j.dental.2015.04.015
- Chen, C., Niu L. N., Xie, H., Zhang, Z. Y., Zhou, L. Q., Jiao, K., . . . Tay, F. R. (2015). Bonding of universal adhesives to dentine Old wine in new bottles? *Dental Materials*, 43, 525-536.
- Cardenas, A. M., Siqueira, F., Hass, V., Malaquias, P., Gutierrez, M. F., Reis, A., ... Loguercio, A. (2017). Effect of MDP-containing silane and adhesive used alone or in combination on the long-term bond strength and chemical interaction with lithium disilicate ceramics. J Adhes Dent, 19(3), 203-212. doi:10.3290/j.jad.a38414

[254]

**Proceedings of RSU International Research Conference (2020)** Published online: Copyright © 2016-2020 Rangsit University



- Ceballos, L., Camejo, D. G., Victoria Fuentes, M., Osorio, R., Toledano, M., Carvalho, R. M., & Pashley, D. H. (2003). Microtensile bond strength of total-etch and self-etching adhesives to caries-affected dentine. *J Dent*, 31(7), 469-477. doi:10.1016/s0300-5712(03)00088-5
- Da Silva, L. H., De Lima, E., Paula Mirando, R. B., Favero, S. S., Lohbauer, U., & Cesar, P. F. (2017). Dental ceramics: a review of new materials and processing methods. *Braz Oral Res*, 31(58), 133-146.
- De Munck, J., Mine, A., Van den Steen, P. E., Van Landuyt, K. L., Poitevin, A., Opdenakker, G., & Van Meerbeek, B. (2010). Enzymatic degradation of adhesive-dentin interfaces produced by mild selfetch adhesives. *Eur J Oral Sci*, 118(5), 494-501. doi:10.1111/j.1600-0722.2010.00758.x
- Derbanne, M. A., Besse, V., Le Goff, S., Sadoun, M., & TN., P. (2014). The effect of functional monomer chain spacer length on the bond strength of an experimental dental adhesive. *International Journal of Adhesion and Adhesives*, 55, 95-105.
- Giannini, M., Makishi, P., Ayres, A. P. A., Vermelho, P. M., Fronza, B. M., Nikaido, T., & J, T. (2015). Self-etch adhesive systems: a literature review. *Braz Dent J*, *26*(1), 3-10.
- Gracis, S., Thompson, V. P., Ferencz, J. L., Silva, N. R., & Bonfante, E. A. (2015). A new classification system for all-ceramic and ceramic-like restorative materials. *Int J Prosthodont*, 28(3), 227-235. doi:10.11607/ijp.4244
- Gunal, B., & Ulusoy, M. M. (2017). Optical properties of contemporary monolithic CAD-CAM restorative materials at different thicknesses. *J esthet restor dent*, *30*, 434-441.
- Guth, J. F., Almeida, E., Silva, J. S., Ramberger, M., Beuer, F., & Edelhoff, D. (2012). Treatment concept with CAD/CAM-fabricated high-density polymer temporary restorations. *J Esthet Restor Dent*, 24(5), 310-318. doi:10.1111/j.1708-8240.2011.00497.x
- He, L. H., & Swain, M. (2011). A novel polymer infiltrated ceramic dental material. *Dent Mater*, 27(6), 527-534. doi:10.1016/j.dental.2011.02.002
- Hori, S., Minami, H., Minesaki, Y., Matsumura, H., & Tanaka, T. (2008). Effect of hydrofluoric acid etching on shear bond strength of an indirect resin composite to an adhesive cement. *Dent Mater J*, 27(4), 515-522. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/18833764
- Hoshika, S., Kameyama, A., Suyama, Y., De Munck, J., Sana, H., & Van Meerbeek, B. (2018). GPDMand 10-MDP- based self etch adhesives bonded to bur-cut and uncut enamel-"Immediate" and "aged" MTBS. *J Adhes Dent*, 20(2), 113-120.
- Ikeda, M., Tsubota, K., Takamizawa, T., Yoshida, T., Miyazaki, M., & Platt, J. A. (2008). Bonding durability of single-step adhesives to previously acid-etched dentin. *Oper Dent*, 33(6), 702-709. doi:10.2341/08-26
- Isolan, C. P., Valente, L. L., Münchow, E. A., Basso, G. R., Pimentel, A. H., Schwantz, J. K., ... R. R., M. (2014). Bond strength of a universal bonding agent and other contemporary dental adhesives applied on enamel, dentin, composite, and porcelain. *Applied Adhesion Science*, 2(25). Retrieved from https://appliedadhesionscience.springeropen.com/articles/10.1186/s40563-014-0025-x
- Iwai, H., & Nishiyama, N. (2012). Effect of calcium salt of functional monomer on bonding performance. J Dent Res, 91(11), 1043-1048.
- Kalavacharla, V., Lawson, N., Ramp, L., & Burgess, J. (2014). Influence of etching protocol and silane treatment with a universal adhesive on lithium disilicate bond strength. *Oper Dent*, 40, 372-378.
- Lise, D. P., Van Ende, A., De Munck, J., Vieira, L., Baratieri, L. N., & Van Meerbeek, B. (2017). Microtensile bond strength of composite cement to novel CAD/CAM materials as a function of surface treatment and aging. *Oper Dent*, 42(1), 73-81. doi:10.2341/15-263-L
- Mamanee, T., Takahashi, M., Nakajima, M., Foxton, R. M., & Tagami, J. (2015). Initial and long-term bond strengths of one-step self-etch adhesives with silane coupling agent to enamel-dentincomposite in combined situation. *Dent Mater J*, 34(5), 663-670. doi:10.4012/dmj.2015-050
- Matinlinna, J. P., & Lassila, L. V. (2010). Experimental novel silane system in adhesion promotion between dental resin and pretreated titanium. Part II: effect of long-term water storage. *Silicon*, *2*, 79-85.
- Moszner, N., Salz, U., & Zimmermann, J. (2005). Chemical aspects of self-etching enamel-dentin adhesives: a systematic review. *Dent Mater*, 21(10), 895-910. doi:10.1016/j.dental.2005.05.001

[255]

**Proceedings of RSU International Research Conference (2020)** Published online: Copyright © 2016-2020 Rangsit University



- 1 MAY 2020
- Munoz, M. A., Luque-Martinez, I., Malaquias, P., Hass, V., Reis, A., Campanha, N. H., & A. D., L. (2015). In vitro longevity of bonding properties of universal adhesives to dentin. *Oper Dent*, 40(3), 282-292.
- Murillo-Gomez, F., Rueggeberg, F. A., & De Goes, M. F. (2017). Short- and long-term bond strength between resin cement and glass-ceramic using a silane-containing universal adhesive. Oper Dent, 42(5), 514-525. doi:10.2341/16-211-L
- Noda, Y., Nakajima, M., Takahashi, M., Mamanee, T., Hosaka, K., Takagaki, T., . . . Tagami, J. (2017). The effect of five kinds of surface treatment agents on the bond strength to various ceramics with thermocycle aging. *Dent Mater J*, 36(6), 755-761. doi:10.4012/dmj.2016-383
- Oliveira, B. M. B., Ulbaldini, A. L. M., Sato, F., Baesso, M. L., Bento, A. C., Andrade, L. H. C., . . . Pascotto, R. C. (2017). Chemical interaction analysis of an adhesive containing 10-Methacryloyloxydecyl Dihydrogen Phosphate (10-MDP) with the dentin in noncarious cervical lesions. *Oper Dent*, 42(4), 357-366. doi: 10.2341/16-062-L
- Pashley, D. H., Tay, F. R., Breschi, L., Tjäderhane, L., Carvalho, R. M., Carrilho, M., & Tezvergil-Mutluay, A. (2011). State of the art etch-and-rinse adhesives. *Dent Mater*, 27(1), 1-16. doi: 10.1016/j.dental.2010.10.016
- Perdigao, J., Kose, C., Mena-Serrano, A. P., De Paula, E. A., Tay, L. Y., Reis, A., & Loguercio, A. D. (2014). A new universal simplified adhesive: 18-month clinical evaluation. *Oper Dent*, 39(2), 113-127.
- Peumans, M., Valjakova, E. B., De Munck, J., Mishevska, C. B., & Van Meerbeek, B. (2016). Bonding effectiveness of luting composites to different CAD/CAM materials. J Adhes Dent, 18(4), 289-302. doi:10.3290/j.jad.a36155
- Ruse, N. D., & Sadoun, M. J. (2014). Resin-composite blocks for dental CAD/CAM applications. *J Dent Res*, 93(12), 1232-1234. doi:10.1177/0022034514553976
- Sarr, M., Kane, A. W., Vreven, J., Mine, A., Van Landuyt, K. L., Peumans, M., . . . De Munck, J. (2010). Microtensile bond strength and interfacial characterization of 11 contemporary adhesives bonded to bur-cut dentin. *Oper Dent*, 35(1), 94-104. doi:10.2341/09-076-L
- Soares, C. J., Soares, P. V., Pereira, J. C., & Fonseca, R. B. (2005). Surface treatment protocols in the cementation process of ceramic and laboratory-processed composite restorations: A Literature Review. J Esthet Restor Dent, 17(4), 224-235.
- Spitznagel, F. A., Horvath, S. D., Guess, P. C., & Blatz, M. B. (2014). Resin bond to indirect composite and new ceramic/polymer materials: a review of the literature. J Esthet Restor Dent, 26(6), 382-393. doi:10.1111/jerd.12100
- Stawarczyk, B., Liebermann, A., Eichberger, M., & Guth, J. F. (2015). Evaluation of mechanical and optical behavior of current esthetic dental restorative CAD/CAM composites. *J Mech Behav Biomed Mater*, 55, 1-11. doi:10.1016/j.jmbbm.2015.10.004
- Strasser, T., Preis, V., Behr, M., & Rosentritt, M. (2018). Roughness, surface energy, and superficial damages of CAD/CAM materials after surface treatment. *Clin Oral Investig*, 22(8), 2787-2797. doi:10.1007/s00784-018-2365-6
- Sturz, C. R., Faber, F. J., Scheer, M., Rothamel, D., & Neugebauer, J. (2015). Effects of various chair-side surface treatment methods on dental restorative materials with respect to contact angles and surface roughness. *Dent Mater J*, 34(6), 796-813. doi:10.4012/dmj.2014-098
- Terry, D. A., & Blatz, M. B. (2010). Surface treatments for tooth-colored restorations: part I. Dent Today, 29(8), 108-110. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/20873655
- Terry, D. A., & Blatz, M. B. (2011). Surface treatments for tooth-colored restorations: Part 2. *Dent Today*, 30(3), 126, 128, 130-121. Retrieved from https://www.ncbi.nlm.nih.gov/pubmed/21485890
- Tian, F., Wang, X., Huang, Q., Niu, L., Mitchell, J., Zhang, Z., . . . Tay, F. (2016). Effect of nanolayering of calcium salts of phosphoric acid ester monomers on the durability of resin-dentin bonds. *Acta Biomaterialia*, 38, 190-200.
- Tian, T., Tsoi, J. K., Matinlinna, J. P., & Burrow, M. F. (2014). Aspects of bonding between resin luting cements and glass ceramic materials. *Dent Mater*, 30(7), e147-162. doi:10.1016/j.dental.2014.01.017

# [256]

**Proceedings of RSU International Research Conference (2020)** Published online: Copyright © 2016-2020 Rangsit University



- Tsujimoto, A., Barkmeier, W. W., Takamizawa, T., Wilwerding, T. M., Latta, M. A., & Miyazaki, M. (2017). Interfacial characteristics and bond durability of universal adhesive to various substrates. *Oper Dent*, 42(2), e59-e70. doi:10.2341/15-353-L
- Van Landuyt, K. L., Snauwaert, J., De Munck, J., Peumans, M., Yoshida, Y., Poitevin, A., . . . Van Meerbeek, B. (2007). Systematic review of the chemical composition of contemporary dental adhesives. *Biomaterials* 28, 3757-3785.
- Van Landuyt, K. L., Yoshida, Y., Hirata, I., Snauwaert, J., De Munck, J., Okazaki, M., . . . B., V. M. (2008). Influence of the chemical structure of functional monomers on their adhesive performance. *J Dent Res*, 87(8), 757-761.
- Van Meerbeek, B., Van Landuyt, K., & De Munck, J. (2005). Technique-sensitivity of contemporary adhesives. *Dent Mater J*, 24(1), 1-13.
- Van Meerbeek, B., Yoshihara, K., Yoshida, Y., Mine, A., De Munck, J., & Van Landuyt, K. L. (2011). State of the art of self-etch adhesives. *Dent Mater*, *27*, 17-28.
- Wagner, A., Wendler, M., Petschelt, A., Belli, R., & Lohbauer, U. (2014). Bonding performance of universal adhesives in different etching modes. J Dent, 42, 800-807.
- Wang, R., Shi, Y., Li, T., Pan, Y., Cui, Y., & Xia, W. (2017). Adhesive interfacial characteristics and the related bonding performance of four self-etching adhesives with different functional monomers applied to dentin. J Dent, 62, 72-80. doi:10.1016/j.jdent.2017.05.010
- Yao, C., Yu, J., Wang, Y., Tang, C., & Huang, C. (2018). Acidic pH weakens the bonding effectiveness of silane contained in universal ashesives. *Dent Mater*, 34, 809-818.
- Yoshida, K., Kamada, K., & Atsuta, M. (2001). Effects of two silane coupling agents, a bonding agent, and thermal cycling on the bond strength of a CAD/CAM composite material cemented with two resin luting agents. J Prosthet Dent, 85(2), 184-189. doi:10.1067/mpr.2001.113628
- Yoshida, Y., Yoshihara, K., Nagaoka, N., Hayakawa, S., Torii, Y., Ogawa, T., . . . Van Meerbeek, B. (2012). Self-assembled nano-layering at the adhesive interface. *J Dent Res*, 91(4), 376-381.
- Yoshida, Y., Nagakane, K., Fukuda, R., Nakayama, Y., Okazaki, M., Shintani, H., . . . Van Meerbeek, B. (2004). Comparative study on adhesive performance of functional monomers. *J Dent Res*, *83*(6), 454-458.
- Yoshihara, K., Nagaoka, N., Okihara, T., Kuroboshi, M., Hayakawa, S., Maru, OY., . . . Van Meerbeek, B. (2015). Functional monomer impurity affects adhesive performance. *Dent Mater*, *31*, 1493-1501.
- Yoshihara, K., Yoshida, Y., Hayakawa, S., Nagaoka, N., Irie, M., Ogawa, T., . . . Van Meerbeek, B. (2011). Nanolayering of phosphoric acid ester monomer on enamel and dentin. *Acta Biomater*, *7*, 3187-3195.
- Yoshihara, K., Yoshida, Y., Hayakawa, S., Nagaoka, N., Torii, Y., Osaka, A., . . . Van Landuyt, K. L. (2011). Self-etch monomer-calcium salt deposition on dentin. *J Dent Res*, 90(5), 602-606.

[257]