



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

Rasita Kusolphat

**A Thesis Submitted in Partial Fulfillment of the Requirements for the
Degree of Doctor of Philosophy in Oral Health Sciences**

Prince of Songkla University

2024

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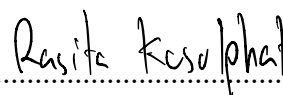


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ชื่อวิทยานิพนธ์	ความแข็งแรงยึดเหนี่ยวของเนื้อฟันกับวัสดุบูรณะชนิดต่างๆเมื่อยึดด้วยซีเมนต์เรซินแบบยูนิเวอร์ซัล
ผู้เขียน	นางสาวรสิตา กุศลพัฒน์
สาขาวิชา	วิทยาศาสตร์สุขภาพช่องปาก
ปีการศึกษา	2566

บทคัดย่อ

วัตถุประสงค์: เพื่อศึกษาถึงความแข็งแรงยึดเหนี่ยวและลักษณะความล้มเหลวบริเวณพื้นผิวยึดติดระหว่างเนื้อฟันกับวัสดุบูรณะชนิดต่างๆเมื่อยึดด้วยซีเมนต์เรซินแบบยูนิเวอร์ซัล 3 ชนิด

วัสดุและวิธีการทดลอง: ใช้ฟันกรามซี่ที่สามของมนุษย์จำนวน 40 ซี่ ทำการตัดตามความยาวฟันโดยฟันแต่ละซี่จะถูกแบ่งได้เป็น 4 ส่วน จากนั้นทำการจับ 10 ชิ้นเข้ากลุ่ม โดยแบ่งตามชนิดของวัสดุบูรณะและซีเมนต์เรซินแบบยูนิเวอร์ซัล ขึ้นตัวอย่างมีลักษณะเป็นทรงกระบอกหน้าตัด 2 มิลลิเมตร ทำการขึ้นรูปด้วยวัสดุบูรณะที่แตกต่างกัน 5 ชนิด ได้แก่ โลหะผสมนิกเกิลโครเมียม ทอง กลาสเซรามิกชนิดลิเทียมไดซิลิเกต เซอโคเนียม และเรซินแมทริกซ์เซรามิก จากนั้นทำการยึดวัสดุบูรณะกับเนื้อฟันตามที่บริษัทแนะนำด้วยซีเมนต์เรซินแบบยูนิเวอร์ซัล 3 ชนิด ได้แก่ Panavia V5, Duo-Link Universal และ RelyX Universal หลังจากนั้นนำชิ้นตัวอย่างผ่านเครื่องจำลองการเปลี่ยนแปลงอุณหภูมิและนำไปทดสอบความแข็งแรงยึดเหนี่ยวด้วยเครื่องทดสอบชนิดสากลและตรวจดูลักษณะของความล้มเหลวด้วยเครื่องวัดความหนาผิวชิ้นงานแบบ 3 มิติ นำชิ้นตัวแทนหลังทดสอบส่องดูพื้นผิวแตกหักด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด (SEM) แล้วนำข้อมูลที่ได้มาวิเคราะห์ผลทางสถิติ

ผลการทดลอง: กลาสเซรามิกชนิดลิเทียมไดซิลิเกตแสดงค่าความแข็งแรงยึดเหนี่ยวสูงที่สุด 36.14 เมกะปาสคาลเมื่อทำการยึดด้วยซีเมนต์ชนิด Panavia V5 ในขณะที่โลหะผสมนิกเกิลโครเมียมให้ค่า

ความแข็งแรงยึดเหนี่ยวสูงที่สุด 38.02 เมกะปาสกาลเมื่อทำการยึดด้วยซีเมนต์ชนิด Duo-Link Universal ซึ่งซีเมนต์ทั้ง 2 ชนิดนี้ให้ค่าความแข็งแรงยึดเหนี่ยวไม่แตกต่างกันเมื่อทำการยึดโลหะผสม นิกเกิลโครเมียม เซอโคเนียม และกลาสเซรามิกชนิดลิเทียมไดซิลิเกต ($p>0.05$) ในขณะที่เมื่อทำการยึดทองและเรซินแมทริกซ์เซรามิกจะให้ค่าความแข็งแรงยึดเหนี่ยวที่ต่ำกว่า นอกจากนี้หากทำการยึดด้วยซีเมนต์ชนิด RelyX Universal จะให้ค่าความแข็งแรงยึดเหนี่ยวที่ไม่แตกต่างกันในทุกวัสดุบูรณะ ($P>0.05$) ลักษณะของความล้มเหลวที่พบมากที่สุดคือ การล้มเหลวที่ผิวยึดระหว่างวัสดุบูรณะและซีเมนต์เรซิน รองลงมาคือ การล้มเหลวที่ผิวยึดระหว่างเนื้อฟันกับซีเมนต์เรซิน

สรุปผลการทดลอง: ทองและเรซินแมทริกซ์เซรามิกให้ค่าความแข็งแรงยึดเหนี่ยวต่ำเมื่อเทียบกับวัสดุชนิดอื่นๆ ในขณะที่โลหะผสมนิกเกิลโครเมียม เซอโคเนียม และกลาสเซรามิกชนิดลิเทียมไดซิลิเกตให้ค่าความแข็งแรงยึดเหนี่ยวสูงเมื่อยึดด้วย Panavia V5 และ Duo-Link Universal ส่วน RelyX Universal ให้ค่าความแข็งแรงยึดเหนี่ยวต่ำกว่าซีเมนต์เรซินชนิดอื่นๆและไม่พบความแตกต่างเมื่อใช้ยึดวัสดุบูรณะชนิดต่างๆ

นัยสำคัญทางคลินิก ชนิดของซีเมนต์เรซินแบบยูนิเวอร์ซัลและวัสดุบูรณะมีผลต่อค่าความแข็งแรงยึดเหนี่ยวของเนื้อฟัน โดย Panavia V5 และ Duo-Link Universal มีประสิทธิภาพในการยึดติดกับโลหะผสมนิกเกิลโครเมียม เซอโคเนียม และกลาสเซรามิกชนิดลิเทียมไดซิลิเกต ในขณะที่ RelyX Universal ซึ่งเป็นซีเมนต์เรซินแบบยูนิเวอร์ซัลชนิดที่มีสารยึดติดอยู่ในตัวเอง (self-adhesive) ให้ค่าความแข็งแรงยึดเหนี่ยวที่ต่ำกว่า สำหรับพื้นผิวทองและเรซินแมทริกซ์เซรามิกควรมีการใช้ไพรเมอร์เฉพาะเพิ่มเติม

คำสำคัญ: ซีเมนต์เรซิน สารยึดติดเนื้อฟัน สารยึดติดทางทันตกรรม ไพรเมอร์ ความแข็งแรงยึดเหนี่ยว

Thesis Title	Shear bond strength of dentin bonded to various restorative materials using universal resin cements
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Major Program	Oral Health Sciences
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ABSTRACT

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

Materials and Methods: Forty human third molars were longitudinally cut into 4 pieces. Ten sectioned were assigned into a group classified by bonding substrate materials and universal resin cement used. Two-mm diameter cylindrical rods were fabricated from five materials; Nickel-Chromium, Gold, Lithium disilicate glass ceramic, Zirconia, and Resin matrix ceramic. Three resin cements; Panavia V5 (PV5), Duo-Link Universal (DuL), RelyX Universal (ReX) were used to bond cylindrical specimens to prepared dentin surfaces according to the manufacturer's instructions. After subjected to thermocycling, shear bond strength was measured using a universal testing machine, and failure modes were assessed with a 3d profilometer. The representative fracture surfaces were examined using a scanning electron microscope (SEM). The bond strength data were statistically analyzed.

Results: Lithium disilicate glass ceramic displayed the highest bond strength of 36.14 MPa when bonded with Panavia V5, while Nickel-Chromium exhibited the highest bond

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

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

Clinical Significance: The dentin shear bond strength was affected by the types of universal resin cement and substrate materials. Panavia V5 and Duo-Link Universal cement were effective for bonding Nickel-Chromium, Zirconia, and Lithium disilicate glass ceramic while self-adhesive type of universal resin cement, Rely X Universal provided the inferior bond strength. For Gold and Resin matrix ceramic surface, specific primers might be additionally applied.

Keywords: Universal resin cement; Dentin bonded; Restorative materials; Additional primers; Shear bond strength

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LIST OF ABBREVIATIONS AND SYMBOLS

10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate

VBATDT = 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithiol

Ni-Cr = nickel-Chromium

Zr = zirconium

LDS = lithium disilicate

RMC = resin matrix ceramic

PV5 = Panavia V5

DuL = Duo-Link Universal

ReX = RelyX Universal

Al_2O_3 = aluminium oxide

HF = hydrofluoric acid

MPa = megapascal

MMA = methyl methacrylate

HEMA = hydroxyethyl methacrylate

MPTMS = γ -methacryloxypropyl trimethoxysilane

mm = millimeter

μm = micrometers

CAD/CAM = computer-aided design/computer-aided manufacturing

SEM = scanning electron microscope

LIST OF ABBREVIATIONS AND SYMBOLS

LED = light-emitting diode

mw/cm² = milliwatts per square centimetre

°C = degrees Celsius

ADC = adhesive failure between dentin and resin cement

ASC = adhesive failure between restorative material and resin cement

CD = cohesive failure in dentin

CC = cohesive failure in resin cement

CS = cohesive failure in restorative material

MCD = mixed failure at the resin cement-dentin interface

MCS = mixed failure at the resin cement-restorative material interface

MA = mixed failure at the resin cement-restorative material interface and the resin cement-dentin interface

CHAPTER 1

INTRODUCTION

BACKGROUND AND RATIONALE

The popularity of resin cements has increased in recent years. Bonding efficiency of resin cements depends on many factors such as substrate surface treatment or types of resin cement.¹⁻³ There are three types of resin cement classified by substrate modification mechanism; etch-and-rinse, self-etch, and self-adhesive resin cement. The later system is currently widespread used due to their simplicity in bonding procedures. However, many researchers found that the self-adhesive resin cement provide inferior bond strength compared to other systems especially in the case that pre-surface treatments were not achieved.^{4,5}

To improve bonding capability of resin cement to each dental substrate, different surface treatments have been established. Metal primer application was found to be effective for bonding to metal substrates.^{6,7} Hydrofluoric acid etching followed by silane application improved the bond strength to glass-based ceramic surface.^{8,9} Application of MDP primer enhanced the adhesion between resin cement and zirconia surface as well as tooth surface.¹⁰⁻¹² Therefore, the contemporary resin cement systems are presently including the additional primers for tooth or/and

restorative substrate application prior cementation. These cements are claimed to be universal resin cement systems.

Some previous studies have compared the bond strength of these new cement systems to enamel, dentin,^{13,14} and dental restorative substrates.¹⁵⁻¹⁷ However, most studies focused only one bonding interface between resin cement to each substrate, and generally built testing block with resin composite after cement application. However, bonding interfaces between dentin and various substrates using resin cement are complex.^{18,19} With the improvement of resin cements and surface treated primers, it could be wondered that which interface actually obtains inferior bond strength and initiates the failure of the restoration. The research evaluating bond strength of dentin to various restorative material substrates using novel universal resin cements systems under aging simulation should be performed to clarify the problem of cemented restoration including noble metal, base metal, glass ceramic, zirconia and resin-matrix ceramic substrates. The results would be a useful clinical guideline for selection cementation procedures for each dental restorative material.

REVIEW OF THE LITERATURES

Overview of dental resin cement

Resin cements are the most favorable luting agents for indirect restoration due to high retentive strength. Methyl methacrylate-based resin cements have been available since 1952 for cementation of indirect restoration.¹ Conventional Bis-GMA resin cement was modified by adding a phosphate ester to the monomer component that have a degree of chemical bonding as well as a micromechanical bonding to tooth structure and base metal alloys.² There are several characteristics of resin cements that make them clinically superior luting agents. They have good properties such as low solubility, high compressive and tensile strength, favorable aesthetic qualities and presenting micromechanical interlocking to tooth structure. The disadvantages of resin cements are associated with their technique sensitivity, high film thickness and high price. Their bond quality depends on substrate modification systems and compositions. Resin cements may be classified according to their polymerization mechanisms into light-cured, chemical cured and dual-cured.³ The dual-cured resin cements are capable of being cured by means of both light and chemicals. However, the studies have shown that these dual-cure resin cements still require light-curing to reach a high degree of polymerization.²⁰ Resin cements can also be classified by their adhesive scheme into three systems as follows³:

1. Etch-and-rinse resin cements: This system uses a phosphoric acid etching to tooth surface and rinse it off. This etching procedure removes the smear layer and dentinal tubules are opened. Adhesive agent is then applied to the etched surface. The adhesive flows into porosities creating enamel resin tag. In dentinal tissue, acid generate micropores in collagen. Therefore, the adhesion depends on the hybridization of adhesive within exposed collagen. This system provides highest bond strength but also requires many steps to bond. Examples include Variolink II (Ivoclar Vivadent, Amherst, NY, USA), Calibra (Dentsply Caulk, Milford, DE, USA).^{21,22}
 2. Self-etch resin cements: This system uses self-etching primer to modify tooth surface without washing process, then air blowing to remove the solvent. Adhesive agent is then applied to the prepared surface. This system reduces technique sensitivity and increases ease of use. Examples include Panavia F2.0, Panavia V5 (Kuraray America, New York, NY, USA)
 3. Self-adhesive resin cements: This system is currently popular due to one-step use. They do not require etching, primer or bonding agents to bond to tooth surface. Bond strengths vary widely with this system. Bonding quality rely on functional acidic monomers in their components. Examples include RelyX Unicem, RelyX Unicem 2 Automix (3M ESPE), Duo-Link Universal (BISCO)
- Three systems of resin cements have different tooth surface pretreatment techniques. Some studies reported that etch-and-rinse system provided superior

clinical reliability due to the increasing of bonding surface area for micromechanical retention and forming actual hybrid layer resulting in high retention rates and good marginal seal.^{23,24} Bond strength is a quantitative parameter in order to determine the effectiveness of adhesive systems in enamel and dentin. Some studies showed that conventional or etch-and-rinse systems provided greater shear bond strength than other systems, independently of methodology to test.²⁵ However, the demineralized dentin area with etch-and-rinse system may provide too deep demineralization and become weak point for adhesion due to the incomplete infiltration of resin monomers.^{26,27} This would be the main reason for decreasing bond strength and promoting nanoleakage. Self-etch system can be used in case of tooth sensitivity due to weaker acid creating partial removal of the smear layer without dentinal tubule opening. Novel resin cement has been recently developed to reduce steps of the working procedures. This system is called self-adhesive resin cements. The acidic functional monomers are used to simultaneously etch and chemically bond to tooth substrate without any pretreatment. By reducing the number of steps in bonding procedure, the risk of contamination is less, and technique sensitivity is diminished. However, some studies reported that the bond strength in this system were lower than other systems.^{4,5} This system require enamel etching or selective etching for improving the bond strength.²⁸

Cementation process in some materials requires the application of the specific primers, which are not contained in the resin cement systems, such as noble metal

that requires metal primer or glass ceramic that needs silane application. Nowadays, manufacturer try to produce the resin cement that is claimed to be universal resin cement systems. These novel universal resin cements systems can be used with various dental substrates including base metal, noble metal, glass ceramic, zirconia, and resin-based substrates. These systems contain all the components needed for cementation of indirect restorations such as tooth primer (self-etch primer), restorative primer. Some company utilize one-bottle universal primer for both tooth and restoration surface. Examples include Panavia V5 (Kuraray Noritake Dental, Tokyo, Japan.) and Duo-link universal (kit with All-Bond Universal) (BISCO).^{10,16-18}

The latest version of universal resin cement that utilizes self-adhesive system is RelyX universal (3M ESPE, St Paul, MN, USA). Its bonding performance to dentin was improved by adding amphiphilic adhesion monomer and novel amphiphilic initiator. A highly crosslinked 3D polymer network is formed. To enhance bond strength to all substrates, manufacturer recommends using with 3M Scotchbond Universal Plus adhesive. This adhesive acts as universal primer that contains MDP and silane.

Bond strength of resin cements to restoration and tooth structure

Two important interfaces affect the bond strength of an indirect restoration. Those are the interface between the tooth and the luting resin and interface between the luting resin and inner surface of restoration. Several studies compared shear bond strength of various types of resin cements to tooth structure or restorative materials

including metal alloys, glass-based ceramic and zirconia.^{13,14,29-34} However, most studies conduct the bond strength test by focusing only one interface. The composite blocks or rods are usually used to bond to substrates with tested resin cement. This method is effective for comparison reason on each bonding substrate. However, with the improvement of current resin cement systems, bond strength to various substrate has been enhanced. It is still uncertain that which interface is truly responsible for the failure of the restoration when using this effective resin cement. Investigating two interfaces at the same time may create the difficulty in result explanation due to complex failure type may occur. Therefore, only a few studies conducted the bond strength test of resin cements by bonding restorative materials to dentin.^{18,19} In contrary, lots of previous studies investigated the bond strength of resin cements either to dentin or restorative interfaces. However, it is difficult to compare bond strength among various studies due to different study design.

Surface modification for improving bond strength of resin cements to dentin and restorative materials

Dentin surface

Dentin bonding is complex because it is a wet tissue, composed of hydroxyapatite particles in collagen protein matrix which compromises adhesion. High water content in dentin represents an extra challenge for resin bonding. The water content in dentin is lower on the surface and higher in the adjacency of the dental

pulp. Adhesion is achieved in dentin mainly due to hybridization and integration process which resin monomers are infiltrated into exposed collagen.²⁵

Dentin surface treatments are different in process based on adhesive systems as described above. The main bonding mechanism of dentin substrate is the interlocking between collagen fiber and polymerized resin. In addition, some functional monomers create an additional chemical interaction potentiality with calcium of hydroxyapatite within the hybrid layer such as 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), which seems to improve bond durability.¹ 10-MDP forms a stable calcium-phosphate salt along with a shallow demineralization of the surface.^{35,36}

Yoshida et al investigated the chemically interaction of 3 functional monomers (10-MDP, phenyl-P, 4-MET) with synthetic hydroxyapatite, using x-ray photoelectron spectroscopy and atomic absorption spectrophotometry to test bonding mechanism of mild self-etch adhesives.³⁵ They found that the monomer 10-MDP readily adhered to hydroxyapatite. This bond appeared very stable, as confirmed by the low dissolution rate of its calcium salt in water. The monomer 4-MET was substantially lower and the bonding of the monomer phenyl-P to hydroxyapatite did not appear to be hydrolytically stable. This result was in agreement with Van Landuyt et al, who found that 10-MDP-based primer showed higher bond strength than other primers.³⁶ Therefore, several current resin cement systems include MDP in the separate primer or in resin to improve bonding quality.

Metal Surfaces

Metal alloys can be classified into base and noble metal alloys. The potential locations for chemical bonding and some micromechanical retention between metal surface and adhesive resin are metal oxide layers. Too thick oxide layers will make the bonding failure occur at that oxide layers. However, adhesive resin cannot bond effectively to metal oxide that too thin. Base metal alloys have high free-surface energy due to containing thick oxide layer. Alumina airborne-particle abrasion or sandblasting procedure can roughen surface of metals and also increase surface area and wettability and create mechanical interlocking and greater affinity between adhesive resins and base metal alloys.⁶ Other methods including tin plating, electrolytic etching, tribochemical silica-coating are disadvantageous due to technique sensitivity, time consuming, and special devices requiring.^{1,6} Noble metal alloys have an ineffective oxide layer. The sulfur-containing metal primers was found to promote the bond strength of resin to noble metal alloy.⁷

Various functional monomers have been used for enhancing the bonding of resin cement to metal surfaces. Phosphoric acidic monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) were recommended to use as surface pretreatment for base metal alloy.³⁷ Sulfur-containing monomers were recommended to use as surface pretreatment for noble metal alloy.³⁸ These monomers contain 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithiol (VBATDT), thiophosphoric acid derivatives (MEPS), 6-methacryloyloxyhexyl 2-thiouracil-

5-carboxylate (MTU-6), or 10-methacryloyloxydecyl-6,6-dithiooctanate (MDDT). Some primers such as Alloy Primer (Kuraray Noritake Dental Inc), composed of VBATDA and MDP mixture, and recommended to use for both noble and base metal alloys. V-Primer and Metaltite are also sulfur-containing primers.³⁹ V-Primer is composed of VBATDT in acetone, and Metaltite consists of 6-methacryloxyhexyl 2-thiouracil-5-carboxylate (MTU-6) in alcohol.

For base metal alloys, Shafiei et al investigated the shear bond strength of self-etch and self-adhesive resin cement to base metal alloys (nickel-chromium alloys) with different surface treatment.¹⁵ They found that sandblasting improved the shear bond strength of both resin cements to base metal alloys. The best results achieved with combination of sandblasting and metal primers (5.31 ± 1.21 MPa for Panavia F2.0). The authors suggested that the performance of resin cement depended on chemical composition, not cement system. Resin cements and alloy primers that consist of 10-MDP monomer could bind chemically to metal oxides of base metal alloys.⁴⁰ MDP is an organic ester which can chemically bond to oxide layer created on metal surface through covalent bonds, and also mechanical retention to the sandblasted surface. Another study investigated shear bond strength of indirect composite resin to Ni-Cr alloy using 4 primers and 2 airborne-particle abrasion procedures and found that airborne-particle abrasion with $50 \mu\text{m}$ Al_2O_3 particles improved the bond strength, independent of primer used.⁶ On the contrary, Fonseca et al investigated the effect of metal primers on shear bond strength of resin cements to base metal alloys including

nickel-chromium (NiCr) and commercially pure titanium (CP Ti) alloys.²⁹ They found that alloy primer did not increase the bond strength to CP Ti and significantly decreased adhesive bonding of panavia F to NiCr alloy. Shafiei et al investigated shear bond strength of self-etch (Panavia F2.0) and two self-adhesive (G-Cem and RelyX Unicem) resin cements to base metal alloy receiving different surface treatments.¹⁵ For Panavia F2.0, they found that sandblasted before priming with alloy primer (4.89 ± 1.28 MPa) had higher shear bond strength than sandblasting (2.79 ± 1.11 MPa) or alloy primer (2.73 ± 6.45 MPa) alone. For G-Cem and RelyX unicem, they found that all surface treatments (sandblasted before priming and sandblasting or alloy primer alone) showed similar shear bond strength.

For noble metal alloys, Antoniadou et al investigated the effect of alloy primer which contained VBATDT plus MDP and ED primer which contain only MDP to sandblasted noble metal alloys.⁴¹ They found that alloy primer significantly improved bond strength of resin cement to noble alloys. They stated that VBATDT promoted a strong and durable bond to noble alloys.

In summary, airborne-particle abrasion with Al_2O_3 particles is the most common method used for promoting micromechanical retention between resin cements and metal surfaces including base and noble metal alloys. Chemical bonding between resin cement and metal alloys can be improved with resin cements or primers containing MDP for base metal alloys and sulfur-containing primers for noble metal alloys.

All-ceramic Surfaces

Gracis et al proposed a new classification system for all-ceramic materials into three families: 1. glass-matrix ceramics 2. polycrystalline ceramics, and 3. Resin-matrix ceramics. The criteria used to differentiate ceramic materials are based on the phases present in their composition.⁴²

1. Glass-matrix ceramic surfaces

Glass-matrix ceramics are all-ceramic materials that contain a glass phase.⁴³ This family is further subdivided into three subgroups according to the crystal types:

1. Naturally occurring feldspathic ceramics that contain leucite crystals (for example: IPS Empress; Ivoclar-Vivadent, Schaan, Liechtenstein)
2. Synthetic ceramics that contain lithium disilicate crystals (for example: IPS Empress 2 or IPS e.max; Ivoclar-Vivadent),
- and 3. Glass-infiltrated ceramics that contain alumina, magnesium or zirconia (for example: In-Ceram Alumina, Vita).

Glass-matrix ceramics are frequently used due to their excellent esthetic properties. Because of their brittleness and limited flexural strength, adhesive cementation is required to improve the fracture resistance of ceramic restoration.

A strong resin bond relies on micromechanical interlocking and chemical bonding to ceramic surface. Acid etching with solutions of hydrofluoric acid (HF) was found to be an effective method for treating ceramic surface to obtain micromechanical retention.⁸ HF attacks glass phase of glass-matrix ceramics, causing

micro-retention due to dislodgement of crystal and achieve proper surface texture and roughness. Guarda et al, reported that etching glass-infiltrated ceramic with 10% HF (35 ± 9.6 MPa for RelyX ARC) shows higher bond strength than sandblasting with $50 \mu\text{m}$ Al_2O_3 (26.9 ± 6.9 MPa for RelyX ARC).⁴⁴ For IPS e.max Press, Puppini-Rontani et al suggested that using of 5% HF for 20 seconds (27.8 ± 1.6 MPa for Variolink II) or 2.5% HF for 120 seconds (25.9 ± 1.3 MPa for Variolink II) was enough to properly dissolve the glassy matrix and achieve greater bond strength.⁴⁵

Silane coupling agents provide a chemical covalent and hydrogen bond which is a major factor for a sufficient resin bond to silica-based ceramics. Silanes are bifunctional molecules. One end is methacrylate group that bond to adhesive containing resin and another end forms siloxane bond to OH group on the ceramic surface. Silanization increases wettability of the ceramic surface and should be a clinical step in cementing silica-based ceramics, even with the application of universal adhesive that contains silane in its formulation.⁹ The strength of resin cement bonded to glass ceramic depends primarily on the silanization of etched ceramic surface. Jardeh et al reported that etching with HF acid alone was not sufficient to produce a strong bond with dental ceramics, but when a silane coupling agent was used, the adhesion bond increased.⁴⁶

The proper surface treatment for improving bond strength of resin cements to silica-based ceramics is HF acid etching followed by silane coupling agent. This method

is provided micromechanical interlocking and chemical bonding of resin cements to ceramic surface.

2. Polycrystalline ceramic surfaces

Polycrystalline ceramics are all-ceramic materials that do not contain any glass phase including alumina and zirconia. HF acid could not create proper surface roughness of zirconia surface.⁴⁷ Alumina air abrasion or tribochemical silica coating combined with MDP-containing primer provided satisfactory bond strength compared to the other mechanical and chemical pre-treatments, particularly when MDP-containing cements were used.¹¹ Air-abrasion with 50-110 μm Al_2O_3 for 5-15 seconds at pressure 2-4 bars at distance of 10 mm was able to create irregularities on surface of zirconia and increase bond strength.⁴⁸⁻⁵⁰ MDP or 10-Methacryloyloxydecyl dihydrogen phosphate consists of a phosphate ester group at one end that provide chemical bond to metal oxides in zirconia and a vinyl group at another end that bonds to resin matrix in adhesive agents.¹²

Tanis and Akcaboy investigated the effects of different surface treatment methods and MDP monomer on shear bond strength between zirconia and two different resin cements including MDP containing resin cement (panavia F2.0) and conventional resin cement (variolink II).⁵¹ They found that Panavia F2.0 (4.35 ± 3.11 MPa) presented statistically higher shear bond strength than that of variolink II (1.98 ± 1.54 MPa) in sandblasting alone groups. Tribochemical silica coating group showed higher

shear bond strength than sandblasting alone group. This result was in consistent with previous studies.^{52,53} However, there are many disadvantages of tribochemical silica coating technique due to forming a too thick silica layer, non-homogeneous silica layer or non-stable silica layer. These reasons are in consistent with the study of Qeblawi et al. They reported that tribochemical silica coating with silanation increased initial bond strength between zirconia and resin cement but in long time period, a decrease in bond strength values was observed.⁵⁴ Several previous studies confirmed that MDP-containing zirconia primer applied after airborne abrasion significantly increased shear bond strength to resin cement.^{55,56} Many researchers proofed that MDP-containing primers applied to the surface before cementation were more essential to increase the bond compared to MDP-containing resin cement.^{10,57,58} In addition, Grasel et al investigated effect of alumina sandblasting on bond strength to zirconia using resin cement.¹⁷ For Duo-link Universal, they found that sandblasting before priming with Z-Prime plus (21.2 ± 5.2 MPa) showed significant higher shear bond strength than priming with Z-Prime plus alone (11.1 ± 6.3 MPa).

In summary, primer or resin cement containing MDP applied after sandblasting is the effective surface treatment method to modify zirconia surface. This method increases bond strength between zirconia and resin cement due to increasing in microroughness, surface energy, and surface wettability for flowing of resin cement.

3. Resin-matrix ceramic surfaces

Resin-matrix ceramics are composed predominantly (>50% by weight) of refractory inorganic compounds. Resin-matrix ceramic materials can be divided into three subfamilies, according to their inorganic composition: 1. Resin nanoceramic (for example: Lava Ultimate, 3M ESPE) 2. Glass ceramic in a resin interpenetrating matrix (for example: Enamic, Vita) 3. Zirconia-silica ceramic in a resin interpenetrating matrix (for example: Shofu Block HC, Shofu)

Shofu HC (Shofu, Kyoto, Japan) is a CAD/CAM composite resin material which consists of UDMA, TEGDMA, silica, barium glass, silica powder, micro fumed silica, and zirconium silicate.⁵⁹ Alumina-oxide (Al_2O_3) particles with particle size of 50 μm is the most effective method to roughen the surface independently of the indirect composite used. Additional silane treatment yield in further increased bond strength of resin bonded to indirect composite resin material.⁶⁰

Strasser et al investigated the effects of different surface pretreatment on CAD/CAM materials on surface topography.⁶¹ They found that HF acid etching showed lower surface roughness than sandblasting. Sandblast with 50 μm aluminum oxide particles at a pressure of 1 bar showed sufficient to attain surface roughness. Some resin primers were found to be effective to improve bond strength of resin cement to resin-based CAD/CAM materials.⁵⁹ Silane coupling agent may promote additional chemical bonding as well owing to the bonding to silica fillers. However, primer containing MMA was found to produce stronger bonding.^{59,62}

Bond Strength Test of dental adhesive materials

Bond strength is defined as the initial mechanical load to fracture divided by the simple, geometrically defined, cross-sectional area of the bond. The bond strength test offers the advantages of facile bond testing for rapid screening of adhesive systems and predict the clinical performance of dental adhesives.⁶³ The common laboratory bond strength testing methods are macroshear/microshear, macrotensile/microtensile bond strength test which are static tests. ISO/TS 11405:2003 divided the macro- and micro-bond strength test according to size of bond area. The macro-bond strength test is the test with a bond area larger than 3 mm² while the micro-bond strength test is the test with a bond area much smaller, about 1 mm² or less.

The shear bond strength test gains its high popularity in several studies since no further specimen processing is needed after the bonding procedure. However, cohesive failures in the substrate were frequently observed with new adhesive that obtained better bond strength. The cohesive failure pattern affected the validity of obtained measurement with this test method. Griffith investigated bond strength in brittle material.⁶⁴ He revealed that relationship between bonding area and bond strength of adhesive materials were affected by the quantity of defects that is the larger the specimen, the greater the number of defects. When the specimen is loaded, stress concentration occurs at the defects and initiates crack formation. This result agreed with Phrukkanon et al, they investigated the effect of bonding area including 1.2, 1.4 or 2 mm in diameter on micro-shear and micro-tensile bond strength

of dental adhesive agents to dentin.⁶⁵ They found that 2.0 mm. diameter group showed significantly lower bond strengths than the 1.2 mm diameter group in both shear and tensile tests. They indicate that small surface areas of test specimens are associated with higher bond strengths. However, micro-bond strength test has a sensitive technique in fabricating specimens due to very small size of specimens. For this reason, macro-shear or conventional shear bond strength test with small size specimens is proposed to reduce technique sensitive and number of defects in bonding interface, leading to reduce in cohesive failure. Several studies revealed the shear bond strength with this test method, their retentive area in cylindrical shape range from 3 to 7 mm² (2 to 3 mm in diameter).^{15,18,22,27,28}

Effect of thermocycling on bond strength of resin cement

Thermocycling is the condition that most often used to test durability of resin bonds. This is considered clinically relevant aging parameters. D'Amario et al investigated the durability of bond strength between zirconia and 3 different dual-cured resin cements, after aging by thermocycling.⁶⁶ They found that thermocycling played a significant role in some cements in degradation of resin cement/zirconia ceramic bonds. This result was in agreement with Holderegger et al, they found that thermocycling affected bond strength of some resin cements, leading to significantly lower shear bond strength.¹³ Oyague et al stated that a statistically significant decline in bond strength of MDP-containing resin cement after 6 months of water storage when

luting to sandblasted zirconia surface.⁶⁷ Therefore, in vitro laboratory test on bond strength of resin cement should be done with thermocycling for simulating aging condition to provide more clinically relevant results.

Objectives

1. To evaluate the shear bond strengths of dentin bonded to various restorative materials using three contemporary universal resin cements
2. To evaluate the failure mode of the bonding interfaces between dentin and various restorative material substrates using three contemporary universal resin cements

Null hypotheses

1. There was no significant difference in the bond strength of the dentin bonded to various restorative material substrates.
2. There was no significant difference in bond strength among the three universal resin cements used to bond dentin to various restorative material substrates.

CHAPTER 2

RESEARCH METHODOLOGY

RESEARCH DESIGN

Experimental research (Laboratory)

MATERIALS AND METHODS

Summary methodology is presented in the diagram as follows:

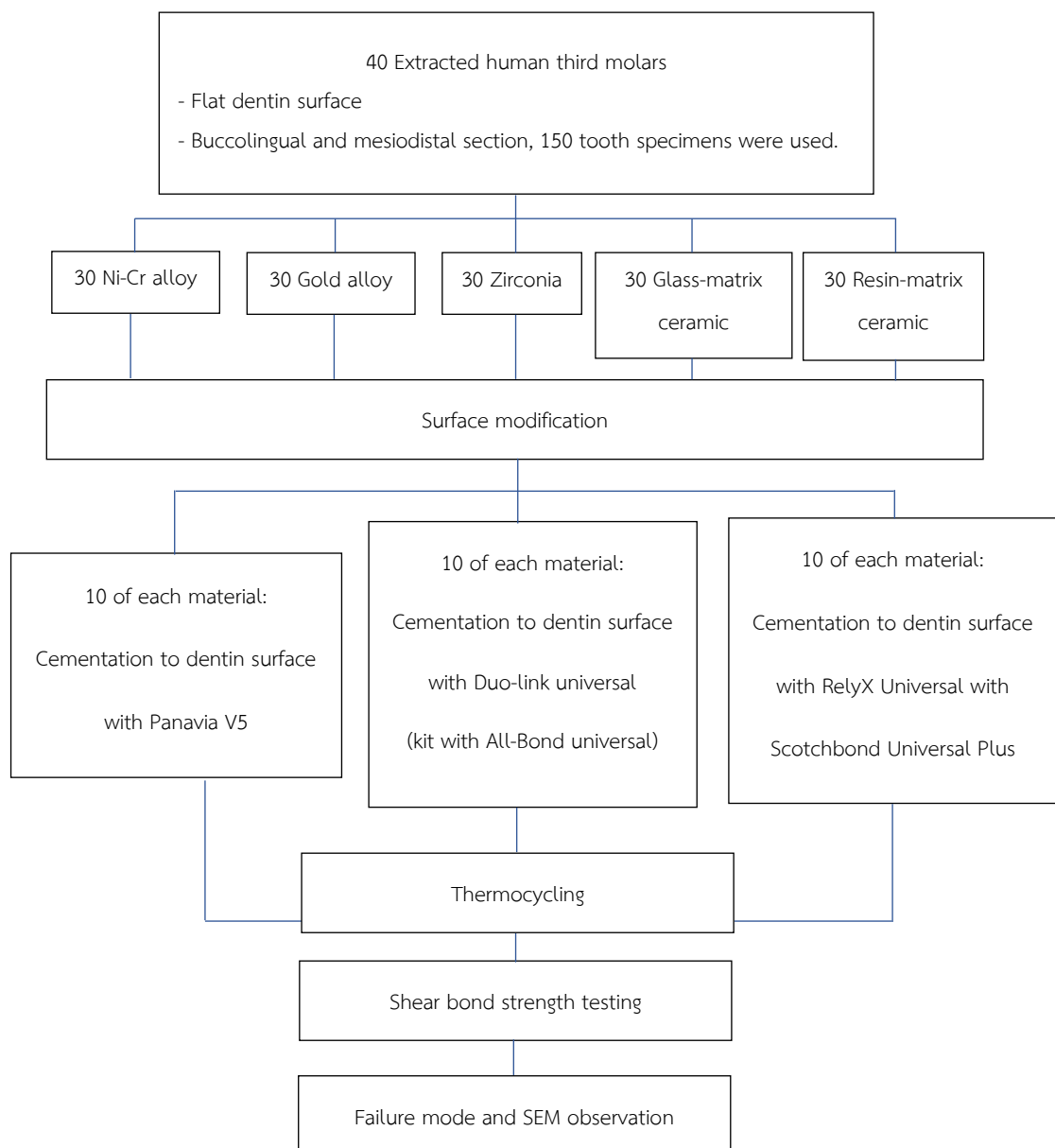


Figure 1. Diagram showing the process of this study

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

Product	Composition	Manufacturer
Argeloy N.P.	Ni 61.2%, Cr 25.8%, Mo11.0%, Si 1.5%, Mn <1%, Al <1%	Argen Co, San Diego, USA
Aurium D69	Au 69%, Pt 4.1%, Pd 1.9%, Ag 14.5%, Cu 9.45%, Zn 1%, Ir <1%	Aurium Research, USA
IPS e.max Press	Lithium disilicate glass ceramic	Ivoclar Vivadent, Schaan, Liechtenstein
Cercon	Zirconia ceramic	Dentsply Sirona, USA
Shofu disk HC	UDMA, TEGDMA, 61 wt% silica powder, micro fumed silica, zirconium silicate	Shofu, Kyoto, Japan
IPS Ceramic Etching Gel	4.5% hydrofluoric acid gel	Ivoclar Vivadent, Schaan, Liechtenstein
Panavia V5	- Tooth primer (self-etching primer): 10-MDP, 2-HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water - Clearfil ceramic primer plus (prosthesis primer): 3-Methacryloxypropyl trimethoxysilane, 10-MDP, ethanol - Paste: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica Bisphenol A, silanated aluminium oxide filler, dl-Camphorquinone, pigments	Kuraray Noritake Dental, Tokyo, Japan
Duo-Link Universal (Kit with All-bond universal)	- All-Bond Universal: 10-MDP, Bis-GMA, HEMA, ethanol, water, initiators - Z-prime plus: Biphenyl dimethacrylate, MDP, ethanol - Porcelain primer: Silane coupling agent - Paste: Bis-GMA, triethyleneglycol dimethacrylate, glass filler	BISCO Inc, Schaumburg, IL, USA
RelyX Universal with Scotchbond Universal Plus	- Scotchbond Universal Plus: Bisphenol A derivative free dimethacrylate monomers including a novel radiopaque monomer, MDP Phosphate Monomer, HEMA hydrophilic monomer, 3M Vitrebond Copolymer, Non-settling silica filler, ethanol, water, photo initiator, optimized mixture of silanes, dual-cure accelerator - Paste: Bisphenol A derivative free dimethacrylate monomers, phosphorylated dimethacrylate adhesion monomers, photo initiator, Novel amphiphilic redox initiator, Radiopaque fillers, rheological additives, pigments	3M ESPE, St Paul, MN, USA
MDP: Methacryloyloxydecyl dihydrogen phosphate; VBATDT: 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithiol; HEMA: Hydroxyethyl methacrylate; Bis-GMA: Bisphenol A diglycidylmethacrylate; TEGDMA: Triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate; MMA: Methyl Methacrylate; MPTMS: γ-methacryloxypropyl trimethoxysilane		

Preparation of dentin surfaces

Forty extracted human third molars were used in present study. All teeth did not present caries, restoration and attrition at occlusal surface and no craze line at all surface. All teeth were cleaned to remove calculus and soft tissues. The teeth were collected and kept in 0.1% thymol solution at room temperature until they were used.

The teeth were mounted up to 1 mm below the cementoenamel junction in self-cured acrylic resin using a plastic tube. The occlusal surface of teeth was cut to obtain flat dentin surface. Each tooth was then cut in buccolingual and mesiodistal dimensions and cut at cementoenamel junction to separate the occlusal surface to four pieces. Cutting procedure was performed by specimen cutting machine with water cooling (Buehler, ISOMET 1000, Buehler Ltd., Lake Bluff, Illinois, USA).

One hundred fifty sectioned teeth were embedded in self-cured acrylic resin using a plastic tube with the flat dentin surface facing upward. The dentin surfaces were finished with 600-grit abrasive paper by using rotary grinding machine (Buehler, Metaserve, Buehler, Lake Bluff, Illinois, USA) to create a uniformly flat surface.

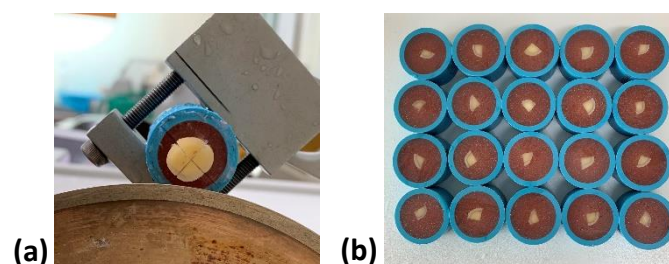


Figure 2. Dentin preparation, (a) Tooth divided into four pieces, (b) Sectioned teeth embedded in self-cured acrylic resin

Preparation of restorative material specimens

Five types of indirect restorative materials as shown in Table 1, thirty pieces of each, were prepared according to the manufacturer's instructions.

Nickel-Chromium alloy, gold alloy and IPS e.max Press specimens were fabricated by conventional lost-wax and casting method using wax pattern created by CAD/CAM technique (Figure 2a). Zirconia and resin-based ceramic specimens were produced by CAD/CAM technique (Figure 2b).

The specimens were cylindrical shape, 2 mm. in diameter and 3 mm. in height. The bonding surfaces of all specimens were finished with 1000-grit abrasive paper by using rotary grinding machine (Buehler, Metaserve, Buehler, Lake Bluff, Illinois, USA) to
(a)
create a uniform surface.

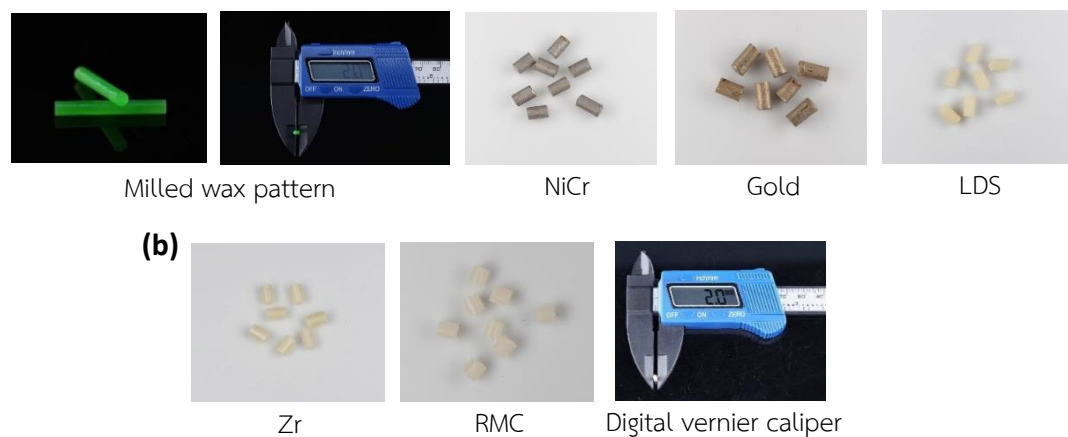


Fig 3. Preparation of restorative material specimens (a) Milled wax and conventional lost-wax for NiCr, Gold, LDS, (b) CAD/CAM technique for Zr and RMC

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

Table 2. Surface treatment of restorative materials before cementation.

Restorative material	Surface treatment procedures
Nickel-Chromium alloy	Airborne-particle abrasion with 50 μm Al_2O_3 for 10 seconds at a distance of 10 mm, a pressure of 2.5 bar perpendicular to surface. (Butler et al, 2018 ¹⁸ , Sarafianou et al, 2008 ⁶ ,)
Gold alloy	
Zirconia ceramic	
Resin-matrix ceramic	Airborne-particle abrasion with 50 μm Al_2O_3 for 10 seconds at a distance of 10 mm, a pressure of 1 bar perpendicular to surface. (Strasser et al, 2018 ⁶¹)
Lithium disilicate glass ceramic	Etching with 4.5% Hydrofluoric acid gel for 20 seconds, rinse with water spray for 5 seconds. (Manufacturer's instructions)

Cementation procedures

Three resin cements, Panavia V5 (universal shade, A2) (Kuraray Noritake Dental, Tokyo, Japan), Duo-Link Universal (universal shade) (BISCO Inc, Schaumburg, IL, USA) and RelyX Universal (translucent shade) (3M ESPE, St Paul, MN, USA) were used to bond each of restorative material to dentin surface. The cements were handled according to the manufacturer's instructions, which were summarized in Table 3.

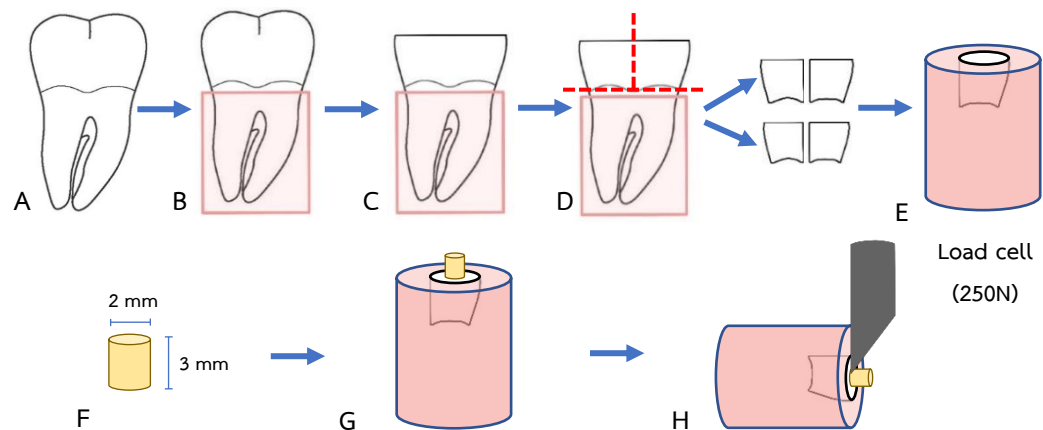


Fig 4. Specimen preparation and testing apparatus for shear bond test. A: Human third molar B: Tooth embedded in acrylic resin. C: Flat occlusal dentin surface. D: Sectioned tooth in 4-parts. E: Specimen embedded in acrylic resin. F: Cylindrical restorative material sample. G: Restorative material bonded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

After completing chemical surface treatment on each surface, the mixture of resin cement was applied onto the restorative material substrates which were then boned to the dentin surface under a pressure of 750 grams for 10 minutes (Gillmore apparatus). Excess cement was removed with microbrush. Light-curing was performed for 20 seconds each from 4 directions by placing the tip of LED light curing (Elipar Trilight; 3M ESPE, St. Paul, MN, USA) close to the bonding interfaces. Light intensity of 800 mw/cm^2 which calibrated by radiometer (Coltolux light meter, Coltene/Whaledent Inc., US) to ensure maximum polymerization of resin cements^{71,72}. The thickness of cement in two representative specimens was measured for each experimental group.

Table 3. Bonding procedures

Resin cement	Substrates	Bonding procedures
Panavia V5	Dentin	- Wash with water spray and dry with air syringe for 10 seconds. - Apply tooth primer with agitating for 20 seconds, dry with air syringe for 5 seconds.
	Restoration	- Apply clearfil ceramic primer plus 10 seconds and dry with air syringe for 5 seconds. - Apply mixed paste to surface of restoration and place restoration on dentin, then remove excess cement with a microbrush. - Light-cured the margin for 20 seconds each from 4 directions.
Duo-link universal (Kit with All-Bond Universal)	Dentin	- Wash with water spray, and use cotton pellets to dry it off - Apply two separate coats of All-bond universal with agitating for 15 seconds per coat. Evaporate excess solvent by strongly air-drying with air syringe for 10 seconds (no visible movement of adhesive). Light cure for 10 seconds.
	Restoration	- Apply 1 coat of Z-prime plus on metal and zirconia surface and dry with air syringe for 3 seconds. - Apply 1 coat of porcelain primer (silane) on lithium disilicate glass and resin-based ceramic surface, wait for 30 seconds, and dry with air syringe for 3 seconds. - Apply mixed paste to surface of restoration and place restoration on dentin, then remove excess cement with a microbrush. - Light-cured the margin for 20 seconds each from 4 directions.
RelyX Universal with Scotchbond Universal Plus	Dentin	- Wash with water spray, and use cotton pellets to dry it off - No additional treatment
	Restoration	- Apply Scotchbond Universal Plus 20 seconds and dry with air syringe for 5 seconds. - Apply mixed paste to surface of restoration and place restoration on dentin, then remove excess cement with a microbrush. - Light-cured the margin for 20 seconds each from 4 directions.



Figure 5. Cementation under a pressure of 750 grams (Gillmore apparatus)

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

Shear bond strength test

After thermocycling, the shear bond strength test was carried out on universal testing machine (Lloyd instruments, Model LRX-Plus, AMETEK Lloyd Instrument Ltd., Hampshire, UK). All specimens were loaded to failure (Figure 6) at a crosshead speed of 0.5 mm/min. with a knife-edge chisel which was placed nearly to bonding interface. The maximum failure load was recorded in newton (N). The shear bond strength values of each specimen were calculated in MPa by dividing the failure load (N) by the bonding area (mm^2) which was πr^2 ; r is radius of bonding interface, 1 mm.

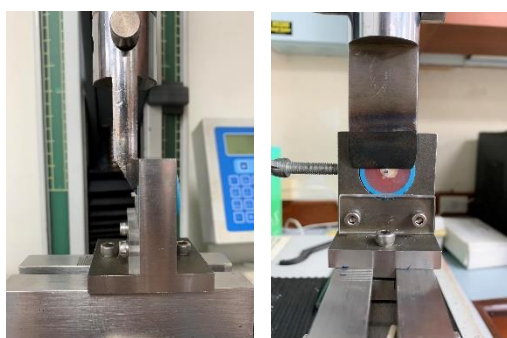


Figure 6. Shear bond strength testing

Determine the mode of failure

After shear bond strength testing, the failure mode was assessed by inspecting the bonding surfaces of each specimen under 3d profilometer (Keyence, VR6200, Keyence Corporation, Japan). Criteria for classification are shown in Table 4. The percentages of each failure modes were defined by ImageJ software. The failure modes are classified into one of the following eight groups as follow:

1. Adhesive failure between dentin and resin cement (ADC)
2. Adhesive failure between restorative material and resin cement (ASC)
3. Cohesive failure in dentin (CD)
4. Cohesive failure in resin cement (CC)
5. Cohesive failure in restorative material (CS)
6. Mixed failure at the resin cement-dentin interface (MCD)
7. Mixed failure at the resin cement-restorative material interface (MCS)
8. Mixed failure at the resin cement-restorative material interface and the resin cement-dentin interface (MA)

Table 4. Criteria to determine each failure mode

Failure mode	Definition
Adhesive failure	Failure occurred equal or more than 70% of entirely bonded area within the bonding interface (dentin-resin cement interface or restorative material-resin cement interface)
Cohesive failure	Failure occurred equal or more than 70% of entirely bonded area within the substrate (dentin, resin cement or restorative material)
Mixed failure	Combination of adhesive and cohesive failure within the one bonding interface (resin cement-dentin interface or resin cement-restorative material interface)

SEM observation of the failure mode

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

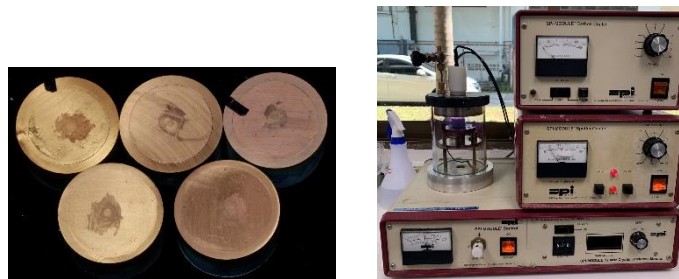


Figure 7. Sputtered specimens with gold in sputter coater (SPI-module)

Statistical analysis

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

CHAPTER 3

RESULTS

The mean shear bond strength and standard deviations for all experimental groups are presented in Table 5. The median of the bond strength values and the significant differences ($p < 0.05$) are shown in Figure 8 and 9.

Table 5. Median shear bond strength values (MPa) (Interquartile range) for all groups

Cement Substrate	Panavia V5	Duolink Universal	RelyX Universal
NiCr	30.23 (19.18) ^{AB,ab}	39.4 (20.31) ^{A,a}	12.74 (5.64) ^{A,b}
Gold	18.76 (4.56) ^{BC,a}	15.3 (6.24) ^{B,a}	13.44 (15.42) ^{A,a}
Zirconia	29.74 (12.95) ^{AB,a}	22.84 (20.93) ^{AB,a}	11.64 (11.37) ^{A,b}
LDS	36.78 (13.38) ^{A,a}	34.08 (21.95) ^{A,a}	12.26 (8.31) ^{A,b}
RMC	10.44 (3.97) ^{C,b}	17.25 (6.89) ^{B,a}	11.82 (5.85) ^{A,ab}

Different uppercase letters indicate significant differences in columns. Different lowercase letters indicate significant differences in rows (Kruskal Wallis and Bonferroni correction, $p < 0.05$). Abbreviation: NiCr=Nickel-Chromium, LDS=Lithium disilicate, RMC = Resin matrix ceramic

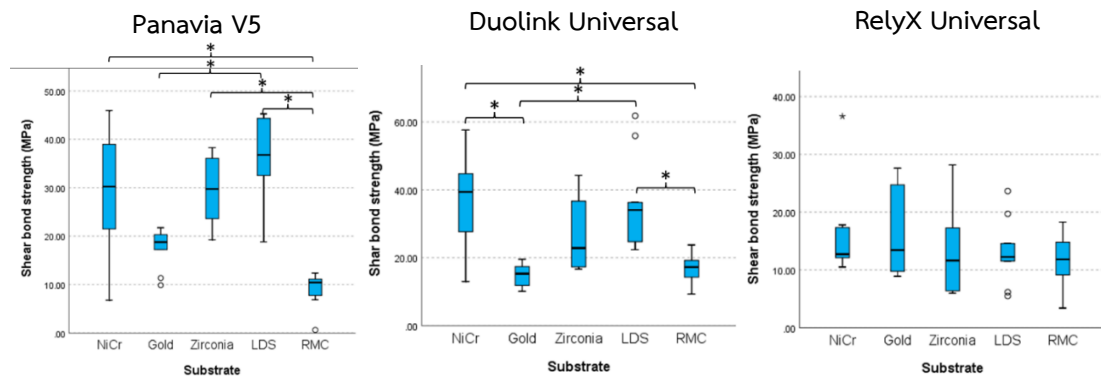


Figure 8. The median of each experimental group when comparing within the same cement A. Panavia V5; B. Duolink Universal; C. RelyX Universal; The asterise (*) indicates significant difference ($p < 0.05$).

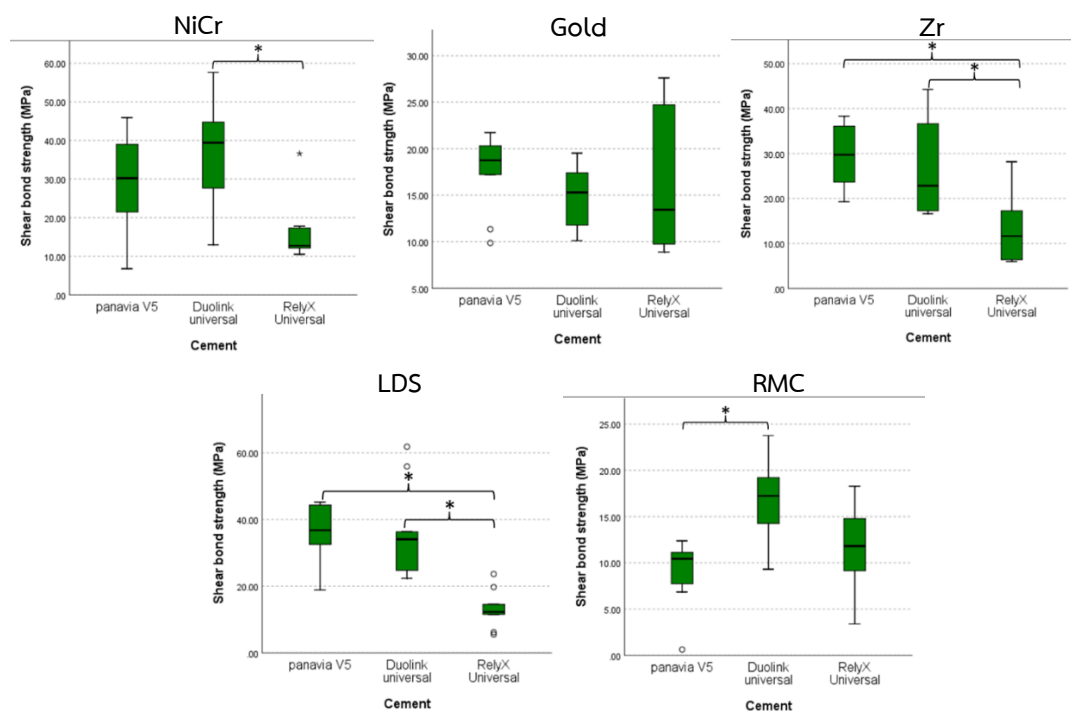


Figure 9. The median of each experimental group when comparing within the same substrate. A. Nickel-Chromium alloy; B. Gold alloy; C. Zirconia ceramic; D. Lithium disilicate glass ceramic; E. Resin-matrix ceramic; The asterise (*) indicates significant difference ($p < 0.05$).

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

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

there was a similar trend in bond strength values. PV5 and DuL provide the comparable bond strength ($P>0.05$), while the bond strength obtained from ReX was significantly lower than those of the two cements. For RMC, DuL demonstrated the highest mean shear bond strength to dentin (16.85 MPa), which was no significant difference from that of ReX (11.64 MPa). Conversely, PV5 exhibited the lowest mean shear bond strength (8.96 MPa) with no significant difference from that of ReX.

For failure mode observation, the frequency distribution of failure modes is presented in Table 6. Representative scanning electron microscope (SEM) images in each failure mode are displayed in Figure 10 - 14. For precious metal, Gold, the failure patterns were mainly occurred at the interface between cement and substrate interface regardless of the cement type. On the other hand, the base metal alloy (NiCr) and RMC demonstrated various type of failure mode depending on the cement used. Cohesive failure in cement including the mixed mode of this failure type frequently observed when PV5 was used. Adhesive failure between substrate and cement was 50% detected for DuL. Almost all of the specimens bonded with ReX failed as adhesive failure between dentin and cement, except for Gold and LDS failed as adhesive failure between cement and substrate. The Zr interfaces predominantly failed as adhesive failure between cement and substrate for PV5 and DuL whereas adhesive failure between dentin and cement occurred for ReX.

Table 6. Number of specimens in each failure mode

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

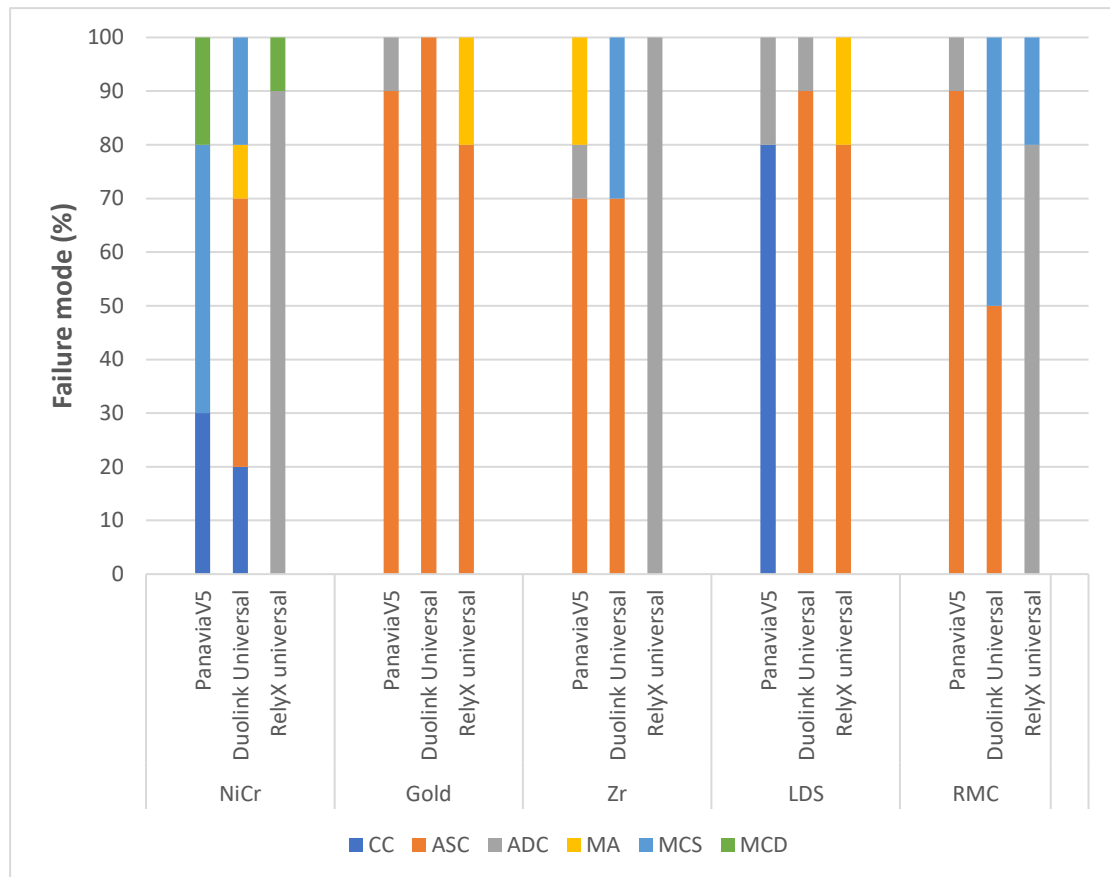


Figure 10. Bar graph of fracture mode proportions. CC: cohesive failure in resin cement; ASC: adhesive failure between restorative material and resin cement; ADC: adhesive failure between dentin and resin cement; MA: mixed failure at the resin cement-restorative material interface and the resin cement-dentin interface; MCS: mixed failure at the resin cement-restorative material interface; MCD: mixed failure at the resin cement-dentin interface.

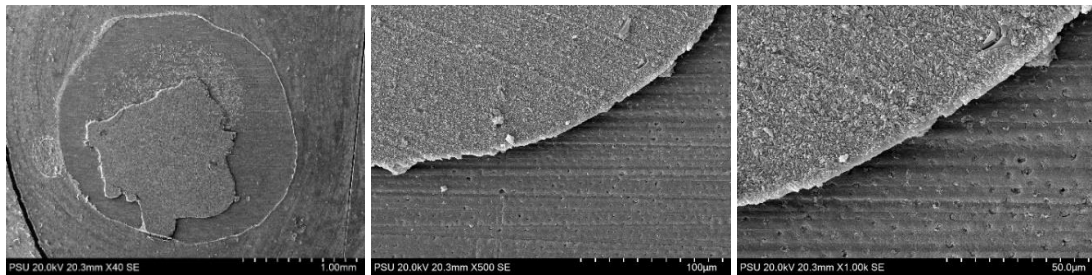


Figure 11. Scanning electron microscope images ($\times 40$, $\times 500$, $\times 1000$ magnification) in dentin site luting between LDS and ReX showing mixed failure at resin cement-restorative material interface and resin cement-dentin interface. Abrasive polished surface of dentin was observed at resin cement-dentin interface.

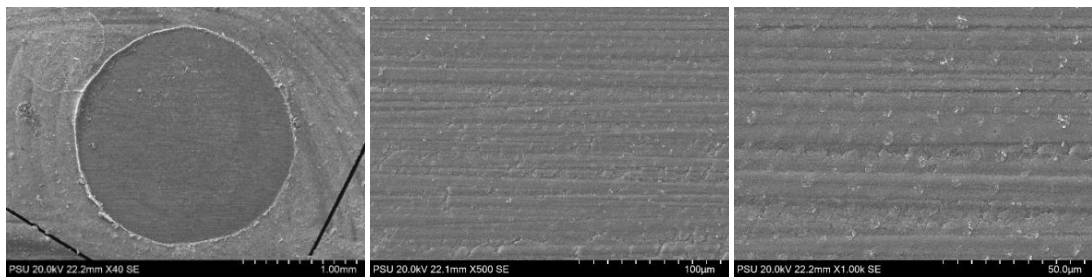


Figure 12. Scanning electron microscope images ($\times 40$, $\times 500$, $\times 1000$ magnification) in dentin site luting between Zr and ReX showing adhesive failure between dentin and resin cement. Abrasive polished surface of dentin was predominantly detected.

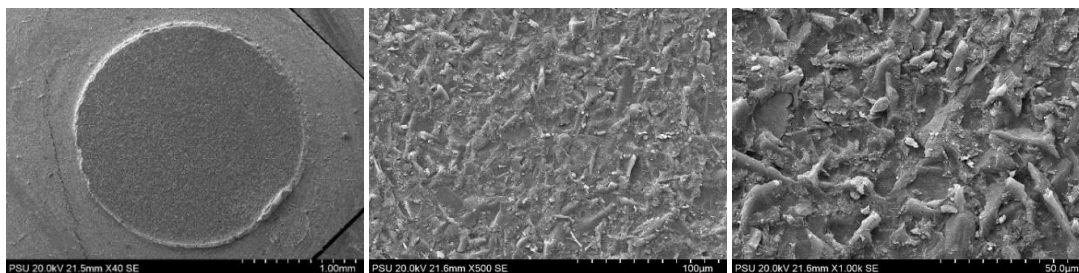


Figure 13. Scanning electron microscope images ($\times 40$, $\times 500$, $\times 1000$ magnification) in dentin site luting between Gold and PV5 showing adhesive failure between restorative material and resin cement. The resin particles covered the dentin surface, and abrasive polished surface of dentin was not observed.

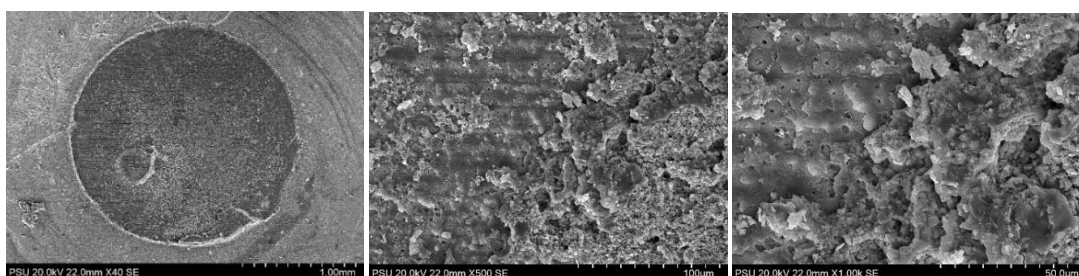


Figure 14. Scanning electron microscope images ($\times 40$, $\times 500$, $\times 1000$ magnification) in dentin site luting between NiCr and PV5 showing mixed failure at resin cement-dentin interface. Abrasive polished surface of dentin and resin cement cluster were simultaneously observed.

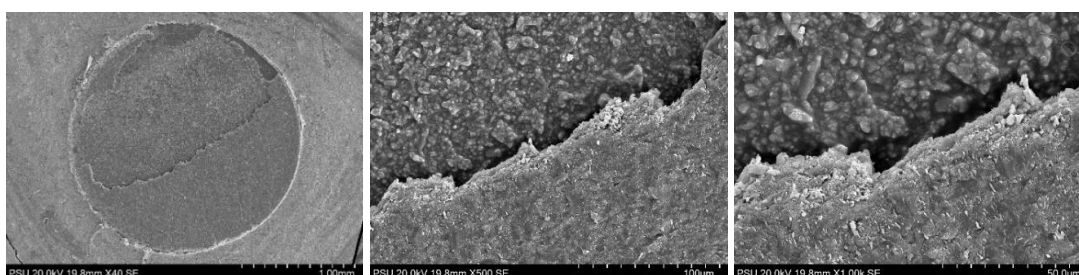


Figure 15. Scanning electron microscope images ($\times 40$, $\times 500$, $\times 1000$ magnification) in dentin site luting between NiCr and PV5 showing mixed failure at resin cement-restorative material interface. Resin cement cluster and substrate surface were simultaneously noticed.

CHAPTER 4

DISCUSSION

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

Thermocycling is the condition most often used to test the durability of resin bonds. This is considered a clinically relevant aging parameter. It was found that thermocycling played a significant role in some resin cements degradation, leading to significantly lower shear bond strength. Several studies reported that thermocycling significantly decreased the bond strength of self-etch and self-adhesive resin cements.^{13,66,68} Therefore, all specimens in the present study were subjected to thermocycling to simulate aging condition to provide more clinically relevant results.

Statistical comparison in some groups revealed no significant difference, even though the bond strength values appeared distinct. The bond strength test is a sensitive method. A defect in the bonding interface generates non-uniform stress

distribution in the defective area, which then initiates crack formation.⁶⁴ The large variation in bond strength values created a non-normal data distribution, a non-parametric statistical analysis was therefore employed in the present study. Consequently, the median was utilized instead of the mean, resulting in the removal of data outliers. As a result, the significant difference was difficult to detect.

In our experimental design, we did not examine the surface roughness of dentin and substrate surfaces after finishing with abrasive paper. This may affect bond strength, so further studies should calibrate the surface roughness after finishing with abrasive paper. Additionally, we controlled the pressure at a 750-gram load during cementation, which may result in unequal cement thickness. However, we clinically ensured control over the bite force. Previous studies also controlled the pressure at a 750-gram load on metal, zirconia, and glass-based ceramic.^{18,29,69} Furthermore, the present study involved a bond strength test that examined two interfaces (resin cement-dentin interface and resin cement-restorative material interface), potentially leading to non-uniform stress distribution in the bond area. And this present study used various restorative materials with different surface treatments according to previous studies that provided the best bond strength for each material.^{6,18,45,61} These factors are likely contributors to the large variation in bond strength values. However, bonding protocol in the present study following manufacturer's instructions.

The shear bond strength tests were performed in several studies since no further specimen processing is needed after the bonding procedure. However, cohesive

failures in the substrate were frequently observed with new adhesives that obtained better bond strength. The previous study indicated that small surface areas of test specimens were associated with higher bond strengths. Nevertheless, micro-shear bond strength testing is a sensitive technique in fabricating specimens due to a very small size of 1-mm diameter specimens. For this reason, a small size shear specimen was proposed in this study to reduce technique sensitivity and the number of defects in the bonding interface, leading to a reduction in cohesive failure. Previous studies recommended the retentive area in cylindrical shape range from 3 to 7 mm² (2 to 3 mm in diameter).^{15,18} In the present study, the retentive area had a diameter of 2 mm. It was observed that the predominant failure mode was adhesive failure, suggesting that the methods used are acceptable.

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

was used. PV5 and DuL are composed of tooth primers, which contain 10-MDP and restorative primers, which contain 10-MDP and silane. The monomer 10-MDP readily adhered to hydroxyapatite. This bond appeared very stable, as confirmed by the low dissolution rate of its calcium salt in water.³⁵ Therefore, several current resin cement systems include MDP in separate primers or in the resin itself to improve dentin bonding quality. 10-MDP also promoted bond strength in base metal alloy and zirconia.^{12,37} While silane coupling agent provide a chemical covalent and hydrogen bond, which was a major factor for a sufficient resin bond to silica-based ceramics.⁹ The results of the present study are in agreement with those of previous studies. It was demonstrated that 10-MDP promoted bond strength in NiCr and Zr. Shafiei et al showed that sandblasting and metal primers containing MDP improved the shear bond strength of both tested resin cements to NiCr.¹⁵ In addition, it was found that sandblasting and priming with Z-Prime plus improved the shear bond strength of Zr when using DuL.¹⁷ Many researchers proved that MDP-containing primers applied to the Zr surface before cementation were more essential to increase the bond compared to MDP-containing resin cement.^{10,57,58}

For LDS, a strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface. Acid etching with solutions of hydrofluoric acid (HF) was found to be an effective method for treating ceramic surfaces to obtain micromechanical retention.⁸ HF attacks the glass phase of glass-matrix ceramics, causing micro-retention due to dislodgement of the crystal and achieving proper

surface texture and roughness. Using of 5% HF for 20 seconds or 2.5% HF for 120 seconds was effective enough to properly dissolve the glassy matrix and achieve greater bond strength.⁴⁵ Silane coupling agents provide a chemical covalent and hydrogen bond, which is a major factor in providing a sufficient resin bond to silica-based ceramics. Etching with HF acid alone was not sufficient to produce a strong bond with dental ceramics, but when a silane coupling agent was used, the adhesion bond increased.⁴⁶ Therefore, it could be noticed from the present results that PV5 and DuL can promote better bond strength for NiCr, Zr, and LDS. Especially for LDS, the bond strength could be as high as 36 MPa for both cements. The predominant failure pattern for these two cements was adhesive failure between the substrate and cement, except in the case of PV5 to LDS, where there was a cohesive failure within the cement. It is noteworthy that their tooth primers were more effective for dentin surface bonding compared to the substrate. In the case of Gold and RMC, the bond strength tended to be lower compared to the other materials. However, no significant difference was observed in the shear bond strength between these two substrates. The most common failure pattern, similar to other substrates, was adhesive failure between the substrate and cement. The previous studies showed that the primer containing VBATDT will promote a strong and durable bond to noble alloys.⁴¹ The primer containing MMA was found to produce stronger bonding to resin-based CAD/CAM materials.^{59,62} It could be noticed that PV5 and DuL do not contain these two specific monomers, resulting in lower bond strengths for Gold and RMC

compared to the other substrates. Several studies revealed that the adhesion of resin cement to noble metal surfaces were very weak without a specific primer.^{38,41} RMC are composed predominantly (>50% by weight) of refractory inorganic compounds. Previous studies compared the shear bond strengths of CAD/CAM composite blocks and composite cements using different primers and found that a primer containing MMA (HC primer) exhibited significantly higher bond strength to CAD/CAM composite blocks.^{59,62,70} Further study should be performed to evaluate the shear bond strength of these two substrate materials when used together with those specific primers.

In comparison to other cements, ReX provided inferior shear bond strength, with no discernible difference seen across all substrates. Contrary to the other two cements, which are self-etch resin cements composed of a separating primer for tooth and substrate surface. ReX is a self-adhesive resin cement that does not require a primer for the tooth, it only includes a single-bottle universal bonding agent for substrate surface treatment. All cements in this study were used according to the manufacturer's instructions. Therefore, the dentin surface in the self-adhesive resin cement group (ReX) did not receive any pre-surface treatment, which differed from the other two cements. Its bonding quality depends on functional acidic monomers in its components, without the need for a separating primer for dentin. The separating acidic primer would enhance dentin wettability before applying the cement paste. However, Scotchbond Universal Plus, a universal primer for ReX, was applied only to the substrate surface and not for dentin pretreatment. This present study reported

that a self-adhesive resin cement (ReX) exhibited lower shear bond strength compared to self-etch resin cements (PV5 and DuL). This result was consistent with previous studies.¹⁹ Several studies reported that etch-and-rinse and self-etch resin cements had significantly higher bond strengths than self-adhesive resin cements.^{4,5,19,71} The weak adhesion at the cement-dentin interfaces was confirmed by the predominant failure mode, which primarily occurs as adhesive failure between dentin and resin cement when ReX was used.

When comparing the bond strength among three cements within the same substrate, it was found that there were no significant differences among the cements when bonding to Gold (ranging from 14.79 MPa – 17.50 MPa). This was because the additional primers in these three cements do not contain a specific primer for Gold, such as VBATDT, as mentioned above. For NiCr, DuL showed the highest mean shear bond strength (38.02 ± 13.79 MPa), and there was no significant difference compared to PV5 (28.62 ± 11.97 MPa). These two cements have separate restorative primers that contain 10-MDP, which is specific for NiCr³⁷. The 10-MDP primer of PV5 and DuL was also the reason for the high bond strength of Zr. Although Gold exhibited rougher surface than Zr (Fig.5,6) but the chemical bond was not as strong as Zr. For LDS, separated ceramic primers (silane coupling agent) were utilized for PV5 and DuL. High bond strengths (36 MPa) were obtained for both cements. Unfortunately, the ReX system does not include a separate ceramic primer. Scotchbond Universal Plus, a universal primer for ReX, contains a mixture of various agents, including silane,

dimethacrylate monomer, MDP phosphate monomer, and others, which results in a lower quantity of silane compared to the other two cements that have pure silane in their separating primers. Therefore, the bond strength of ReX was relatively low. In the case of RMC, DuL showed the highest mean shear bond strength (16.85 ± 4.76 MPa), while PV5 showed the lowest mean shear bond strength (8.96 ± 3.60 MPa). However, there was no significant difference in ReX. This can be explained by the fact that PV5 has only one bottle of a restorative primer, which is a mixture of MDP and silane, whereas DuL has two separate restorative primers that isolate MDP and silane. Therefore, the silane coupling agent in DuL exhibited greater efficacy compared to that in PV5. In the present study, Shofu HC is zirconia-silica ceramic in a resin interpenetrating matrix. The preceding systematic review and meta-analysis revealed that sandblasting had been advocated as the preferred pretreatment for CAD/CAM hybrid ceramics with high ceramic content, such as Shofu HC. Conversely, pre-treatment with hydrofluoric acid (HF) is recommended for CAD/CAM resin nanoceramics and glass ceramic in a resin interpenetrating matrix, such as Lava Ultimate or Vita Enamic. Nevertheless, both methods were followed by the application of silane to achieve enhanced bond strength.⁷²

The present study compared the shear bond strength of dentin bonded to various restorative materials: Nickel-Chromium, Lithium disilicate glass ceramic, Zirconia, Gold, and Resin matrix ceramic, using three universal resin cements for two interface bonding. Although many studies have evaluated the bond strength of resin

cement to restorative materials or dentin,^{13,14,18,19,29-34} very few have examined two interface bonding,^{18,19} and no study has compared all restorative materials.

Summarily, the present results indicated that type of universal resin cement and substrates affect the bond strength of dentin-resin cement interfaces. The self-etch universal resin cement seemed to be effective enough to bond Nickel-Chromium, Lithium disilicate glass ceramic and Zirconia surface with the accompanied primers in the cement set. However, Gold and Resin matrix ceramic surface may need additional specific primers. This should be further investigated. For the self-adhesive resin cement used in this study, the result showed unreliable bond strength values. The bonding methodology for improving this type of cement should be examined.

CHAPTER 5

CONCLUSION

Within the limitations of this study, it can be concluded that

1. The dentin shear bond strength was affected by the types of universal resin cement and substrate materials.
2. Nickel-Chromium, Zirconia, and Lithium disilicate glass ceramic surfaces showed similar high shear bond strength when Panavia V5 and Duolink Universal resin cement were used, whereas Gold and Resin matrix ceramic surfaces exhibited lower shear bond strength compared to the other materials.
3. RelyX Universal resin cement showed lower shear bond strength than other cements, and there were no significant differences among all substrates.
4. The failure patterns mainly occurred at the interface between cement and substrate. Except for RelyX universal resin cement, almost all of the specimens failed as adhesive failure between dentin and cement.

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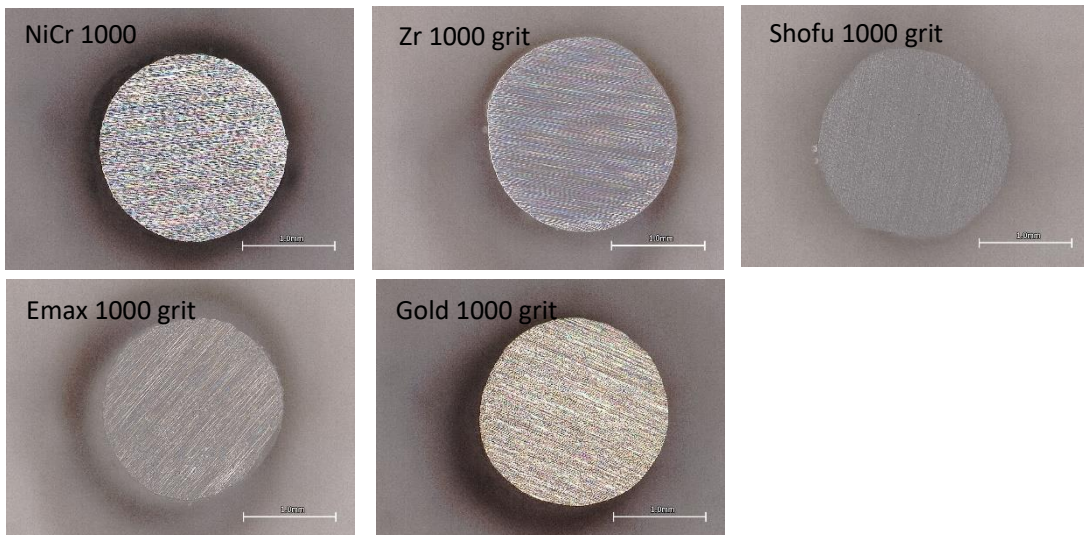
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APPENDIX

Preparation of dentin surfaces



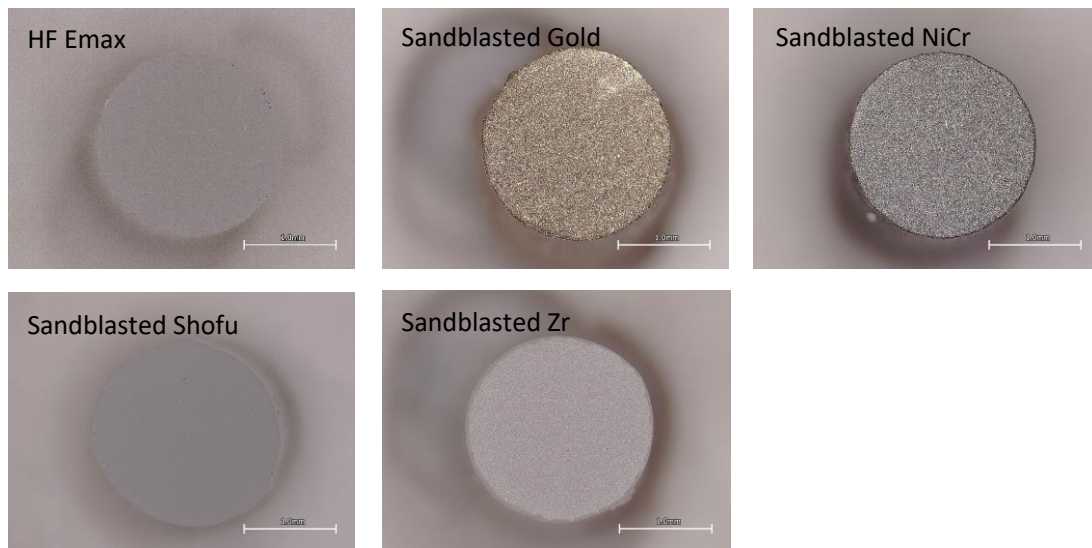
Preparation of restorative material specimens



Mean shear bond strength values (MPa) (standard deviations) for all groups

Cement Substrate	Panavia V5	Duolink Universal	RelyX Universal
NiCr	28.62 (11.97)	38.02 (13.79)	16.03 (7.73)
Gold	17.50 (3.88)	14.79 (3.59)	16.11 (7.48)
Zirconia	29.44 (6.98)	27.07 (11.06)	12.76 (7.10)
LDS	36.14 (8.42)	36.47 (13.87)	13.10 (5.79)
RMC	8.96 (3.60)	16.85 (4.76)	11.64 (4.55)

Surface treatment of restorative materials before cementation



Results of Dunn's post hoc test for Panavis V5 and various substrate

Sample 1-Sample 2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj. Sig. ^a
Resin matrix ceramic-Noble metal	9.633	6.565	1.467	.142	1.000
Resin matrix ceramic-Base metal	23.033	6.565	3.508	.000	.005
Resin matrix ceramic-Zirconia	25.333	6.565	3.859	.000	.001
Resin matrix ceramic-LDS	31.833	6.565	4.849	.000	.000
Noble metal-Base metal	13.400	6.390	2.097	.036	.360
Noble metal-Zirconia	-15.700	6.390	-2.457	.014	.140
Noble metal-LDS	-22.200	6.390	-3.474	.001	.005
Base metal-Zirconia	-2.300	6.390	-.360	.719	1.000
Base metal-LDS	-8.800	6.390	-1.377	.168	1.000
Zirconia-LDS	-6.500	6.390	-1.017	.309	1.000

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same. Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

a. Significance values have been adjusted by the Bonferroni correction for multiple tests.

Results of Dunn's post hoc test for Duolink Universal and various substrate

Sample 1-Sample 2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj. Sig. ^a
Noble metal-Resin matrix ceramic	-3.200	6.390	-.501	.617	1.000
Noble metal-Zirconia	-15.200	6.390	-2.379	.017	.174
Noble metal-LDS	-24.189	6.565	-3.684	.000	.002
Noble metal-Base metal	25.000	6.390	3.912	.000	.001
Resin matrix ceramic-Zirconia	12.000	6.390	1.878	.060	.604
Resin matrix ceramic-LDS	20.989	6.565	3.197	.001	.014
Resin matrix ceramic-Base metal	21.800	6.390	3.412	.001	.006
Zirconia-LDS	-8.989	6.565	-1.369	.171	1.000
Zirconia-Base metal	9.800	6.390	1.534	.125	1.000
LDS-Base metal	.811	6.565	.124	.902	1.000

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same. Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

a. Significance values have been adjusted by the Bonferroni correction for multiple tests.

Results of Dunn's post hoc test for NiCr and three universal resin cement

Sample 1-Sample 2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj. Sig. ^a
RelyX Universal-panavia V5	8.600	3.937	2.184	.029	.087
RelyX Universal-Duolink universal	13.600	3.937	3.454	.001	.002
panavia V5-Duolink universal	-5.000	3.937	-1.270	.204	.612

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same. Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

a. Significance values have been adjusted by the Bonferroni correction for multiple tests.

Results of Dunn's post hoc test for Zr and three universal resin cement

Sample 1-Sample 2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj. Sig. ^a
RelyX Universal-Duolink universal	10.800	3.937	2.744	.006	.018
RelyX Universal-panavia V5	13.950	3.937	3.544	.000	.001
Duolink universal-panavia V5	3.150	3.937	.800	.424	1.000

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same. Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

a. Significance values have been adjusted by the Bonferroni correction for multiple tests.

Results of Dunn's post hoc test for LDS and three universal resin cement

Sample 1-Sample 2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj. Sig. ^a
RelyX Universal-Duolink universal	12.222	3.878	3.152	.002	.005
RelyX Universal-panavia V5	14.356	3.780	3.798	.000	.000
Duolink universal-panavia V5	2.133	3.780	.564	.572	1.000

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same. Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

a. Significance values have been adjusted by the Bonferroni correction for multiple tests.

Results of Dunn's post hoc test for RMC and three universal resin cement

Sample 1-Sample 2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj. Sig. ^a
panavia V5-RelyX Universal	-4.431	3.857	-1.149	.251	.752
panavia V5-Duolink universal	-11.456	3.647	-3.141	.002	.005
RelyX Universal-Duolink universal	7.025	3.765	1.866	.062	.186

Each row tests the null hypothesis that the Sample 1 and Sample 2 distributions are the same. Asymptotic significances (2-sided tests) are displayed. The significance level is .05.

a. Significance values have been adjusted by the Bonferroni correction for multiple tests.

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