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เซอร์โคเนียออกไซด์ต่างชนิดกัน

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MICROTENSILE BOND STRENGTH OF DIFFERENT TYPES OF RESIN CEMENT TO
DIFFERENT ZIRCONIUM-OXIDE CERAMICS

Miss Putsadee Srisomboonkamon

A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Prosthodontics

Department of Prosthodontics

Faculty of Dentistry

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ผุสดี ศรีสมบุญกมล : กำลังยึดดึงระดับจุลภาคของเรซินซีเมนต์ต่างชนิดต่อเซรามิก เซอร์โคเนียมออกไซด์ต่างชนิดกัน (MICROTENSILE BOND STRENGTH OF DIFFERENT TYPES OF RESIN CEMENT TO DIFFERENT ZIRCONIUM-OXIDE CERAMICS) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: ผศ.ทพญ.ดร.ปรารมภ์ ชาลิมี, 68 หน้า.

การศึกษานี้มีวัตถุประสงค์เพื่อศึกษากำลังยึดดึงระดับจุลภาคของการใช้เซรามิกเซอร์โคเนียมออกไซด์ต่างชนิดกันและเรซินซีเมนต์ต่างชนิดกัน โดยใช้เซรามิกเซอร์โคเนียมออกไซด์จาก 3 ชนิดได้แก่ เซอร์คอน ลาวา และคาตานะ และเรซินซีเมนต์ 3 ชนิด ได้แก่ พานาเวียเอฟสอง ซุปเปอร์บอนด์ ซีแอนด์บี และ รีลาย เอ็กซ์ยูนิเซม โดยขึ้นรูปชิ้นงานเซรามิกขนาด $5 \times 5 \times 10$ มิลลิเมตร เป็นจำนวน 3 ชิ้นต่อบริษัท แล้วนำชิ้นงานที่ได้ มาขัดด้วยกระดาษขัดซิลิกอนคาร์ไบด์ หลังจากนั้นนำชิ้นงานไปผ่านกระบวนการเป่าทรายด้วยอนุภาคของอลูมินัมออกไซด์ ขนาด 50 ไมโครเมตรที่แรงดันประมาณ 0.4-0.5 เมกะปาสคาล แล้วจึงนำชิ้นงานทั้งหมดไปทำความสะอาดด้วยอัลตราโซนิกนาน 10 นาที จากนั้นทำการยึดติดกับเรซินคอมโพสิต ขนาด $5 \times 5 \times 10$ มิลลิเมตร ที่ผ่านการขัดด้วยกระดาษขัดซิลิกอนคาร์ไบด์และทำความสะอาดด้วยอัลตราโซนิกเช่นกัน ทำการยึดด้วยเรซินซีเมนต์ ตามคำแนะนำของแต่ละผู้ผลิต จากนั้นนำชิ้นงานที่ถูกยึดติดแล้วมาตัดด้วยเครื่องตัดความเร็วสูง โดยให้ชิ้นงานมีขนาดพื้นที่หน้าตัด 1 ± 0.1 ตารางมิลลิเมตร แล้วนำไปทดสอบกำลังยึดดึงด้วยเครื่องทดสอบสากล ที่ความเร็ว 1 มิลลิเมตรต่อนาที จนชิ้นงานเกิดความล้มเหลวซึ่งสามารถมองเห็นได้ด้วยตาเปล่า นำตัวอย่างที่แตกหักออก นำไปตรวจสอบประเภทของความล้มเหลวโดยใช้กล้องจุลทรรศน์แบบส่องกราด

ผลการศึกษาจากการทดสอบทางสถิติโดยใช้การวิเคราะห์ความแปรปรวนสองทาง พบว่าค่ากำลังยึดดึงของกลุ่ม ลาวา/ซุปเปอร์บอนด์ (53.88 N/mm^2) มีค่าสูงที่สุด ส่วนกลุ่มเซอร์คอน/พานาเวีย (43.28 N/mm^2) มีค่ากำลังยึดดึงระดับจุลภาคที่น้อยที่สุด สรุปได้ว่าชนิดของเรซินซีเมนต์และชนิดของเซรามิกเซอร์โคเนียมออกไซด์มีผลต่อกำลังยึดดึงระดับจุลภาค โดยที่ทั้งสองตัวแปรไม่มีอิทธิพลร่วมกัน

ภาควิชาทันตกรรมประดิษฐ์..... ลายมือชื่อนิสิต.....
 สาขาวิชาทันตกรรมประดิษฐ์..... ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์หลัก.....
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PUTSADEE SRISOMBOONKAMON: MICROTENSILE BOND
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PRAROM SALIMEE, Ph.D.68 pp.

The objective of this study was to evaluate the microtensile bond strength (MTBS) of different types of resin cement to different brands of sandblasted zirconium-oxide ceramic. Three brands of zirconium-oxide ceramic fully sintered blocks (Cercon, Lava and Katana), which surfaces were finished with silicon carbide abrasive and sandblasted with 50 μ m aluminumoxide particles, followed by ultrasonic cleaning in distilled water for 10 minutes. Zirconium-oxide ceramic were bonded to the same size of resin composite blocks (Filtek Z250) that were finished with the same -grit silicon carbide abrasive and cleaned for 10 minutes in an ultrasonic bath. The ceramic-composite blocks were luted with three different resin cements (Panavia F2, Superbond C&B and RelyX Unicem) according to the manufacturer's instructions. The ceramic-composite blocks were cut under water coolant to produce microbar specimens, which have bonding area $1 \pm 0.1\text{mm}^2$. The microtensile bond strength was tested with universal testing machine at a crosshead speed of 1 mm/min. The broken microbars were examined using a scanning electron microscope (SEM).

The MTBS of Lava/Superbond group (53.87 N/mm²) produced the highest MTBS, while Cercon/Panavia group (43.28 N/mm²) produced the lowest MTBS. Type of resin cement and type of zirconium-oxide ceramic have effect on MTBS, while the interaction between zirconium-oxide ceramic and types of resin cement was not significant.

Department: Prosthodontics..... Student's Signature.....
Field of Study: Prosthodontics..... Advisor's Signature.....
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CHAPTER I

INTRODUCTION

All-ceramic restorations have become more popular as restorative dental material such as crowns, bridges and abutments for dental implants. Ceramics are able to mimic the color of the natural adjacent tooth by reproducing a similar transmission and reflection of lights to the underlying tissue. Since soft tissue is transparent, when it is illuminated the pulpal tissue, the tooth root and the surrounding bone will affect the spectral reflectance. Not only a favorable esthetic appearance, but also mechanical properties such as improved high fracture toughness, fracture resistance and biocompatibility are the reasons that all-ceramic restorations gain their popularity [1-3].

There are two major categories of all-ceramic materials: silica-based (feldspathic porcelains, leucite-reinforced ceramics, lithium disilicate ceramics) and non-silica-based (alumina, zirconia). The silica/glass-based all-ceramics are more translucent than alumina- or zirconia-based ceramics and therefore have better optical properties. However, they are mechanically weaker and need to be used in conjunction with resin bonding cements. The bonding in silica/glass-based ceramics uses acid etching at the inner surfaces of glassy matrix with hydrofluoric acid followed by the application of a silane coupling agent which is an efficient method for bonding resin composite [4–9]. Silanes are bi-functional compounds that promote chemical bonding between organic (resin cements) and inorganic (silica-based) materials [10]. This is achieved by functional alkoxy groups on the silane molecule bonding to the silica (SiO_2) phase hydroxyl groups ($-\text{OH}$) on the surface of the dental ceramic. Organo-silanes also have a degradable functional group that copolymerizes with the organic matrix of resin cement

[11, 12]. These processes create the chemical bond that is necessary for the successful bonding of resin cement to dental ceramics. Silanes are also responsible for increasing superficial energy and wettability of ceramic surfaces, which enhance both mechanical and chemical bonding. However, neither etching with hydrofluoric acid nor adding silane coupling agent results in an adequate resin bond to some new high-strength ceramics [13, 14]. Particularly, zirconium-oxide ceramics cannot be roughened by hydrofluoric acid etching since some ceramics do not contain a silicon dioxide (silica) phase [15-20]. Similarly, cement adhesion to silica/glass-based all-ceramic is also not favorable because this ceramic presents the same characteristics due to its high crystal content (aluminum oxide: G67wt%; zirconium-oxide: G13wt%) and limited vitreous phase (lanthanum aluminum silicate:G20 wt%) [21]. For this reason, special bonding systems are indicated for these types of ceramics [22].

Since conventional hydrofluoric acid etching does not have any effect on zirconia surfaces, some investigations have described procedures using airborne-particle abrasion (sandblasting) to achieve resin-to-zirconia bonding [19,23-26] or silica coating, that indicated for treating CAD/CAM sintered porcelains which do not contain a silicon-oxide phase [16,23]. Sandblasting is a prerequisite for achieving sufficient bond strength between the resins and high-strength ceramics that are reinforced either with alumina or zirconia [27]. The air abrasion systems rely on air-particle abrasion with different particle sizes ranging from 30 to 250 μm [22, 28]. The abrasive process removes loose contaminated layers and the roughened surface provides some degree of mechanical interlocking or 'keying' with the adhesive property. It can be argued that the increased roughness also forms a larger surface area for the bond. While these mechanisms explain some of the general characteristics of adhesion to roughened surfaces, they may also

introduce physico-chemical changes that affect surface energy and wettability. Such conditioning systems could be applied either at the laboratory or chairside, using large or small size particles. However, there is limited knowledge as to whether micromechanical retention using large or small particle size increases resin bond to high-strength ceramics of different microstructures and chemical compositions. Blasting-pressure of alumina particles coated with silica onto ceramic surfaces (silica coating) produces a silica layer by embedding the particles, rendering the silica-modified surface chemically more reactive compared to the resin through silane coupling agents [24]. Nevertheless, the effects of these conditioning treatments on zirconia–resin bond have not been exactly determined [29-30]. Raquel Castillo OyagÜe et al. (2009) showed that resin–ceramic interfacial longevity depends on cement selection rather than on surface pre-treatment [31].

Although zirconia restorations can be fixed with conventional cements, the choice of resinous luting agents is advocated to achieve reliable bonds with higher retention and better marginal fit [20, 32]. Few luting agents, such as multi-step phosphate monomer-based resin cements (i.e., Panavia 21, Panavia F) have demonstrated satisfactory bonding to zirconium-oxide ceramics [33-34]. The investigation by Kern and Wegner(1998) found that a durable resin bonded to yttrium oxide partially stabilized zirconia ceramic (YPSZ) was achieved only after sandblasting the ceramic and using MDP-containing cements (Panavia) resulting in the highest MTBS values with zirconia and which decreased slightly over 150 days storage with thermal cycling, though this decrease was not statistically significant[19]. The combination of airborne particle abrasion and MDP monomer is the recommended method of bonding to zirconia frameworks. This method can produce good bond strength and bond durability after

thermo-cycling and long term water storage [35-36]. However, Masuhara E et al. (1990) showed that the bond strength between the PMMA resin Superbond C&B and the sandblasted Procera specimens was initially high, but after 150 days storage time with thermocycling, the bond strength decreased statistically significantly. The bond strength of the MDP-containing composite resin Panavia 21 yielded the highest bond strength. Although there was a slight decrease in bond strength over 150 days stored, it was not statistically significant [37]. In contrast De'rand P et al. (2000) pointed out that Superbond C&B had bond strength by far superior to Panavia 21 between the Denzir surface and resin cement [20].

There are many manufacturers of zirconium-oxide ceramics including Cercon (Degudent GmbH, Hanue-Wolfgang, Germany), Lava (3M ESPE, Seefeld, Germany), Katana (Nuvovent, Noritake Dental Supply, Japan), Procera Zirconia (Nobel Biocare, Yorba Linda, USA), Zeno (Wieland Dental, GmbH, Pforzheim, Germany) and In-Ceram YZ Cubes (Vita Zahnfabrik, Bad Sackingen, Germany). Different zirconium-oxide ceramics might have a little difference in compositions but much difference in procedure and process of fabrication. Therefore, some properties might be different and might affect the bonding surface and bond strength of each brand of zirconia. Also, most of the previous research studies have been performed in bonding zirconia using specific brand. So the aim of this study is to evaluate the bond strength of different types of resin cement to different brands of sandblasted zirconium-oxide ceramic by microtensile test.

CHAPTER II

LITERATURE REVIEW

1.1 Zirconia ceramic

Zirconia ceramic is a ceramic material consisting of at least 90% of zirconium dioxide (ZrO_2). Zirconia crystals can be organized in three different patterns. Pure zirconia changes its crystal structure depending on the temperature: at temperatures below 2138 °F (1170 °C) zirconia exists in monoclinic form, at 2138 °F (1170 °C) monoclinic form transforms to tetragonal form which is stable up to 4300 °F (2370 °C). Tetragonal crystal structure transforms to cubic structure at 4300 °F (2370 °C). Structure transformations are accompanied by volume changes which may cause cracking if cooling or heating is rapid and non-uniform.

Additions of some oxides (MgO , CaO , Y_2O_3) to pure zirconia depress allotropic transformations (crystal structure changes) and allow stabilization of either cubic or tetragonal structure of the material at any temperature. The most popular stabilizing addition to zirconia is yttrium oxide (Y_2O_3), which is added and uniformly distributed in proportion of 5.15%. Depending on sintering temperature and other processing parameters, the following forms of stabilized zirconia may be prepared as: Fully stabilized zirconia (FSZ) with cubic crystal structure, Partially stabilized zirconia (PSZ) with mixed structure (cubic+tetragonal), and Polycrystalline tetragonal zirconia (TZP) with metastable tetragonal structure of very fine zirconia grains sintered at low temperature.

Table 1 Chemical composition of magnesia partially stabilized zirconia (MgPSZ) [38]

Property	Value in metric unit		Value in US unit	
Density	5.6 *10 ³	kg/m ³	349.6	lb/ft ³
Modulus of elasticity	350	GPa	50800	ksi
Flexural strength	545	MPa	79000	psi
Compressive strength	1700	MPa	246500	psi
Fracture toughness	6	MPa*m ^{1/2}	6	MPa*m ^{1/2}
Hardness	1100	HV	1100	HV
Thermal expansion (20 °C)	10*10 ⁻⁶	°C ⁻¹	5.6*10 ⁻⁶	in/(in* °F)
Thermal conductivity	2.5	W/(m*K)	17.3	BTU*in/(hr*ft ² *°F)
Specific heat capacity	400	J/(kg*K)	0.1	BTU/(lb*°F)
Max. working temperature	1000	°C	1830	°F
Dielectric strength (AC)	6	KV/mm	150	V/mil

Table 2 Chemical composition of yttrium oxide stabilized polycrystalline tetragonal zirconia (YTZP) [39]

Property	Value in metric unit		Value in US unit	
Density	6.0 *10 ³	kg/m ³	374.5	lb/ft ³
Modulus of elasticity	200	GPa	29000	ksi
Flexural strength	900	MPa	130500	psi
Compressive strength	2000	MPa	290000	psi
Fracture toughness	13	MPa*m ^{1/2}	13	MPa*m ^{1/2}
Hardness	1300	HV	1300	HV
Thermal expansion (20 °C)	10.3*10 ⁻⁶	°C ⁻¹	5.7*10 ⁻⁶	in/(in* °F)
Thermal conductivity	2	W/(m*K)	13.9	BTU*in/(hr*ft ² *°F)
Specific heat capacity	400	J/(kg*K)	0.1	BTU/(lb*°F)
Max. working temperature	1500	°C	2730	°F

Zirconia ceramic has advantages of high flexural strength (900 MPa to 1.2 GPa) and toughness (7 to 8 MPa·m^{-0.5}), high density (up to 380 lb/ft³ [6.1*10³ kg/m³]), low thermal conductivity (10% of that of alumina ceramics), high maximum service temperature (up to 4350 °F [2400 °C]), high chemical resistance, high resistance to molten metal, coefficient of thermal expansion similar to that of cast iron, modulus of elasticity similar to steel, good wear resistance, low coefficient of friction, oxygen ion conductivity (used for oxygen sensors and high temperature fuel cells) and favorable esthetic appearance. Other beneficial properties include good biocompatibility [1-3].

Zirconia has not only a color similar to teeth but is also opaque, this can be an advantage for the technician, when a discolored tooth or a metal post must be covered, a zirconia core allows concealment of any unfavorable aspect. On the contrary, if translucency is absolutely needed, it can be attained with other ceramic materials such as alumina or lithium disilicate. Moreover, some manufacturers make provision for zirconia colored cores in order to enhance esthetic outcomes. Preventive evaluation of natural teeth color and transparency is necessary to select an appropriate all-ceramic system. Moreover, when zirconia ceramic restoration is preferred, a preoperative choice between different colored cores is suggested. Also, the radiopacity of zirconia is very useful for monitoring marginal adaptation through radiographic evaluation, especially when intrasulcular preparation is used.

A review of the literature evaluated all-ceramic restorations' survival rate compared with PFM and all-ceramic groups with zirconia ceramic restorations [40-41]. 5-year survival rate of all-ceramic restorations resulted in 93.3%, while metal-ceramic restorations resulted in 95.6%. All-ceramic restorations on posterior teeth

resulted to have the lowest 5-year survival percentage (84.4%). On the other hand, when compared with other all-ceramic systems, zirconia frameworks showed as the most reliable. The veneering chipping or cracking was the weakest point of zirconia ceramic, while other all-ceramic restorations revealed a percentage of framework fracture. These results are in accordance with clinical indication for all-ceramic restorations which indicate that all-ceramic systems can be used preferably on anterior teeth; only zirconia ceramic showed adequate mechanical resistance for both anterior and posterior restorations [42].

There are several categories of dental ceramics: classification by type (feldspathic porcelain, leucite-reinforced porcelain, aluminous porcelain, alumina, glass-infiltrated spinel, glass-infiltrated zirconia and glass ceramic); classification by use (denture teeth, metal-ceramics, veneers, inlays, crowns, anterior bridges and posterior bridges); classification by processing method (sintering, casting or machining) or classification by substructure material (cast-metal, swaged metal, glass ceramic, CAD-CAM porcelain or sintered ceramic core). This review delivers classification by system of zirconium-oxide ceramic (Cercon system, Lava system and Katana system).

- **Cercon system**

Cercon® blanks are made of yttrium oxide stabilized zirconia (Y-TZP). They are used in fabricating frameworks for fixed prosthetic restorations using Cercon®brain / Cercon® brain expert. They are made of a ceramic oxide material characterized by particularly high strength. Depending on the framework design, Cercon® frameworks can be veneered with ceramics or inserted as fully anatomical restorations.

The Cercon® eye scans dies and models for crowns and bridges using three camera systems as well as a laser, which can precisely map up to 16 units per model. Automatic margin detection enables ease of case design. Coping thickness, margins and occlusion can all be adjusted. With a scan and processing time of less than 60 seconds per unit, and a scanning precision of 20 microns or less, even difficult model geometries, such as undercuts, are easily read. The art software also provides the ability to design a crown to full contour and cutback in specific measured amounts. The Cercon® brain expert CAM system mills zirconia for excellent marginal integrity and increased operating productivity. The Cercon® heat plus is a large capacity sintering furnace that is designed to optimize zirconia sintering and can sinter up to 16-unit bridges with a maximum temperature of 1600°C.

Cercon compositions are zirconium-oxide 93%, yttrium oxide 5%, hafnium oxide < 2%, aluminum oxide and silicon oxide < 1%. The zirconia can be colored in two different shades (white and ivory). Coefficient of thermal expansion is 10.5 mm/m.k (25 -500°C), flexural strength is approximately 1,200 MPa (three-point flexural testing) and modulus of elasticity: 210 GPa.

- **Lava system**

The Lava system uses a CAD/CAM process for the fabrication of zirconia frameworks all-ceramic crowns and bridges, both for anterior and posterior applications. The ceramic framework consists of zirconia supplemented by specially designed overlay porcelain (Lava™ Ceram). The zirconia can be colored in seven different shades. The frameworks are fabricated using CAD/CAM manufacturing techniques (scanning, computer-aided design, computer-aided manufacturing) for pre-sintered zirconia blanks.

The milled framework, whose size has been increased to compensate for the shrinkage during sintering, is sintered in a special high temperature furnace (1360°C–1530°C).

Pre-sintered Zirconia is prepared by three main steps (Figure 1). The Zirconia powder is pressed and pre-sintered. This step is usually performed by the manufacturer. Then, the dental lab mills the pre-sintered blank and sinters the coping or framework to achieve full density. The preparation of the pre-sintered blanks by each manufacturer is different from each other, depending on the zirconia powder source and both the pressing and the pre-sintering conditions selected.

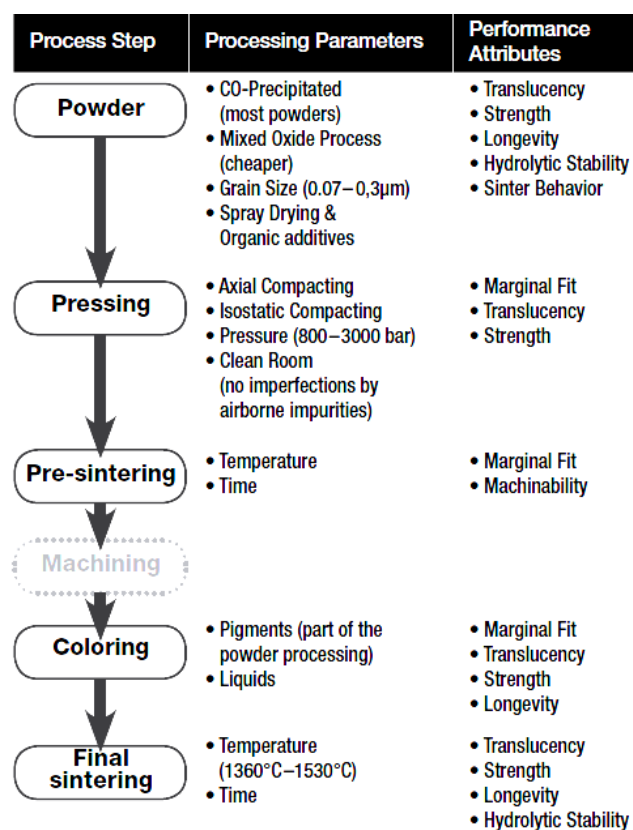


Figure 1 Main steps in the production process of pre-sintered zirconia and the important parameters with their influence on clinical aspects.

The available zirconia powder can have different grain sizes, distributions of the various grain sizes, and additives such as binder for the pressing step. The additives yttrium oxide and alumina can be distributed within the material in a variety of ways such as a homogeneous distribution throughout the whole material, and higher concentration at grain borders. Besides, the grain size has an effect on strength and transformation toughening, a special and key mechanical characteristic of zirconia. Also variations in grain size distribution affect the resulting porosity and hence the translucency of the material. Furthermore, the distribution of additives can affect the hydrothermal stability of the sintered material. So differences in the zirconia powder affect the strength long-term stability and translucency of the restoration.

Also, the powder is pressed, which can be accomplished by different procedures. The pressing conditions are adjusted to get an optimized blank for the pre-sintering step. The pressing methodology influences the homogeneity and the density distribution of the material and hence the marginal fit. The pressing conditions can lead to differences in strength and translucency, and affect the final sintering temperature of the zirconia. The pressed zirconia powder is then pre-sintered in a furnace with an optimized temperature profile to generate a blank with suitable strength and millability. The last step is coloring, which can be achieved either by pigments (grains) or non-pigmented (ions) agents. The zirconia can be colored in seven different shades. It is important to control the effect of the dyeing liquid on the mechanical characteristics of the zirconia material. So coloring of the zirconia can affect the marginal fit, strength and translucency of the material. Some zirconia materials can be colored in the pre-sintered state by immersing copings and frameworks in a dyeing liquid. This enables the absorption of coloring agents in the

zirconia material. Lava compositions are zirconium-oxide 97%, yttrium oxide 3%. Flexural strength is approximately 1,100 MPa.

- **Katana system**

Katana compositions are zirconium-oxide 94.4%, yttrium oxide 5.2% and others. Colors are produced by colored powder, not by dipping, which will affect the stability of color because it is only surface coloring. Pre-colored zirconia frameworks can be colored in nine different shades, using no-presintered zirconia block which has stable shrinkage rate and is easily milled. Coefficient of thermal expansion is 10.5 mm/m.k (50–500°C, flexural strength is approximately 1,000-1,250 MPa.

1.2 Resin cement

Resin cement has become attractive as a luting cement developed for direct-filling resins with improved properties. The benefit of the acid-etch technique is for attaching resins to enamel and the potential to bond to dentin conditioned with organic or inorganic acid. The composition of resin cement is a resin matrix with silane-treated inorganic fillers. The fillers are those used in composites, which are silica or glass particles and/or colloidal silica used in microfilled resins. ISO 4049 for polymer-based filling, restorative, and luting materials (ANSI/ADA No. 27) describes the following three classes of resin cements [43- 44]: Class 1: self-cured materials or chemically activated resin cements are supplied as two-component systems, a powder and a liquid or two pastes. This cement is suitable for all types of prostheses. The excess cement removal is best done immediately after the prostheses are seated. Class 2: light-cured materials are single-component systems just as the light-curable resin-based composites.

This cement is suitable for thin ceramic prostheses, and resin-based prostheses. The required time of exposure to the light for polymerization of the resin cement depends on the intensity of the light transmitted through the ceramic restoration. However, the time of exposure to the light should never be less than 40 seconds. The excess cement should be removed as soon as seating is completed, unless the instructions for the specific resin cement indicate otherwise. Class 3: dual-cured materials are two-component systems. Chemical activation is very low and provides extended working time until the cement is exposed to the curing light. This cement should not be used with light transmitting prostheses which are thicker than 2.5 mm. If the prostheses are thicker than 2.5 mm, they should be bonded chemically with activated resin cements. The excess cement removal may proceed upon completion of seating or after waiting for specific period as indicated in the instructions.

Three types of resin cement were tested in the present study. They were:

- I. PanaviaF 2.0; Kuraray Dental, USA.
- II. Superbond C&B; Sun Medical, Kyoto, Japan.
- III. RelyX™ Unicem; 3M ESPE, St. Paul, MN, USA

I. PanaviaF 2.0

Panavia F2.0 is a self-etching, self-adhesive, dual-cure, fluoride releasing resin cement. The principal ingredients of PanaviaF 2.0 Paste (A Paste/B Paste) are shown in Table 3. The association of airborne-particle abrasion (aluminum oxide or silica-coating) and primer/luting agents containing phosphate ester monomer 10-methacryloyloxydecyl dihydrogenphosphate (MDP) [19,25,26,36,47] and zirconate coupler agent [34,48] has also been suggested. MDP is also present in Panavia F and the phosphate ester group of

this monomer chemically bonds to aluminum and zirconium-oxides [10,19,31,47,49-50]. According to the literature, the presence of MDP in the resin luting agent has been proven to produce good bond strength to airborne particle abraded zirconia and bond durability both before and after thermo-cycling and long term water storage [51]. In previous study, MDP monomer of PanaviaF 2.0 resin cement was successful in establishing good bond strength with zirconia, which was not influenced by 90 days of water storage. The performance of this bond was enhanced by using new types of adhesion promoters designed to enhance wetting and bonding to ceramic substrates [50,52].

Table 3 The principal ingredients of PanaviaF 2.0 Paste (A Paste/B Paste) [45-46].

A Paste	B Paste
<ul style="list-style-type: none"> • 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) • Hydrophobic aromatic dimethacrylate • Hydrophobic aliphatic dimethacrylate • Hydrophilic aliphatic dimethacrylate • Silanated silica filler • Silanated colloidal silica • dl-Camphorquinone • Catalysts • Initiators 	<ul style="list-style-type: none"> • Hydrophobic aromatic dimethacrylate • Hydrophobic aliphatic dimethacrylate • Hydrophilic aliphatic dimethacrylate • Silanated barium glass filler • Surface treated sodium fluoride • Catalysts • Accelerators • Pigments

II. Superbond C&B

Superbond C&B is a self-cure dental adhesive resin cement. The main component is PMMA/MMA. Once polymerized, it becomes PMMA polymer with 4-META (4-methacryloyloxyethyl trimellitate anhydride) as a co-polymer that is structured

as an anhydride with polarity to connect to MMA. When added to PMMA /MMA resin, it enhances the bond strength and "TBB" (tri-*n*-butylborane) as a polymerization initiator. In research publications the Super-Bond system is often referred to as "4-META/MMA-TBB Resin". Superbond C&B was originally introduced in Japan as an orthodontic bonding system called "Orthomite Super-Bond" in 1982. Later, it was introduced in the following year (1983) for general dental adhesive use.

Superbond C&B is classified as an "adhesive resin cement". The manufacturer claims that Superbond contains components that are different from conventional inorganic cements. They also require different clinical handling and their mechanical properties are very different from those of inorganic cements. Most adhesive resin cements consist of polyfunctional dimethacrylate-based monomers, such as Bis-GMA and inorganic fillers of fine glass and silica. In other words, their compositions are similar to that of resin composites. After curing, these composite resin cements form three-dimensional networks of polymerized bi-functional monomers and create a rigid structure with high values in such mechanical properties as micro-hardness and compressive, tensile and flexural strength. But Superbond is different. When it cures, it consists of linear polymers of MMA without inorganic fillers (except for the small pigment traces necessary for shading and for the radio-opacifier in the radiopaque powder.) The resin structure has a microhardness and flexural modulus substantially lower than those of composite resin cements (Table 4).

For example, in one study (Table 4), researchers reported that they were unable to measure Superbond's compressive and tensile strengths because of its low modulus of elasticity, the Superbond samples showed no clear yield-point. Rather than breaking when they were loaded, they displayed high plastic deformation and simply changed

shape. This resilience gives Superbond a significant advantage over traditional adhesive cements. Because the cement remains slightly flexible after curing, it creates a more tenacious bond with higher resistance to occlusal impact stresses.

Table 4 Mechanical properties of luting cements (Yoshida K, et al, 1995)

Cement	Knoop hardness number (KHN)	Compressive strength (MPa)	Diametric tensile strength (MPa)	Flexural strength (MPa)
EC (Zinc phosphate cement)	49.2	124.8	4.4	10.6
HC (Carboxylate cement)	17.3	53.2	5.1	12.4
FB (Glass ionomer cement)	38.4	163.1	10.4	5.5
BR (Resin Cement)	49.8	233.9	34.8	86.5
ID (Resin Cement)	50.0	206.8	45.4	90.0
PE (Resin Cement)	49.6	192.4	32.9	89.6
PT (Resin Cement)	48.9	234.8	34.5	94.4
Super-Bond C&B	8.9	—	—	58.3

—: unable to measure

III. RelyX™ Unicem

Self-adhesive resin cement from 3M ESPE is designed to deliver a strong bond without etching, priming or bonding. The manufacturer revealed that RelyX Unicem cements bind to tooth structure primarily with micromechanical retention which is created by the functional group from their functional monomer, which is phosphate-based. And the secondary retention is from chemical adhesion to hydroxyapatite. The liquid component of RelyX Unicem cement consists of methacrylated phosphoric ester and carbon double bonds(C=C). The inorganic fillers are able to undergo a reaction with the phosphoric acid methacrylates. The reaction starts with free radical polymerization, that can be initiated either by light or redox system (dual-curing composite materials). During polymerization, the reactive carbon double bonds will react with each other and form cross-linked polymers.

Some portions of the filler are silanated and will chemically bond to the methacrylate monomers, while other portions neutralize the phosphoric acid groups of the methacrylate monomers. Water that is released from the reaction will react with the phosphonate group, which will undergo hydrolysis resulting in release of phosphoric acid into the adhesive. The water that has been released will also accelerate the neutralization reaction. The remaining unneutralized phosphoric acid groups will form an ionic bond. The methacrylate monomers and fillers are firmly linked and permanently embedded in the three-dimensional network of the cement matrix.

1.3 Microtensile bond strength

Microtensile bond strength test (MTBS) was introduced by Sano et al. in an effort to develop bond tests of small areas in 1994 [53]. These authors showed that TBS values are inversely related to the bonded surface area [53–55] and that although much higher bond strength values were measured, most failures still occurred at the interface between tooth substrate and adhesive resin. The reason for this increase in bond strength with decrease in bonded surface area is probably due to the presence of defects or stress raisers at the bonded interface or with the substrate. Other advantages of TBS are that regional bond strength and bonding effectiveness could be applied to small sized specimens as a regional area of a tooth substrate focusing on a carious region [56-57] or for example a localized area of sclerotic dentin [58-59] or irregular surfaces. Moreover, the means and variances can be calculated for single teeth or sample. On the other hand, the major disadvantages of MTBS are that the test is rather labor-intensive, technically demanding, and requires careful handling of the fragile specimens. Special care should be taken to reduce the

production of micro-fractures at the interface during specimen preparation which may weaken the bond and thus reduce the actual bond strength [60]. Also the samples, which are small, can dehydrate rapidly.

Objectives

1. To evaluate the microtensile bond strength when different types of resin cement are used.
2. To evaluate the microtensile bond strength when different brands of zirconium-oxide ceramic are used.
3. To evaluate the microtensile bond strength when different types of resin cement to different brands of zirconium-oxide ceramic are used.

Hypotheses

Hypothesis 1

Null hypothesis: There would be no significant difference in microtensile bond strength when using different types of resin cement.

Alternative hypothesis: There would be significant difference in microtensile bond strength when using different types of resin cement.

Hypothesis 2

Null hypothesis: There would be no significant difference in microtensile bond strength when using different brands of zirconium-oxide ceramic.

Alternative hypothesis: There would be significant difference in microtensile bond strength when using different brands of zirconium-oxide ceramic.

Hypothesis 3

Null hypothesis: There would be no significant difference in microtensile bond strength when using different types of resin cement to different brands of zirconium-oxide ceramic.

Alternative hypothesis: There would be significant difference in microtensile bond strength when using different types of resin cement to different brands of zirconium-oxide ceramic.

Keywords

1. microtensile bond strength
2. resin cements
3. zirconium-oxide ceramic.

Proposed benefits

1. To gain informative data for considering the different brands of zirconium-oxide for microtensile bond strength.
2. To gain informative data for considering the clinical use of brands of zirconium-oxide and types of resin cement.

Type of research

Laboratory experimental research

CHAPTER III

MATERIALS AND METHODS

Materials used in this study

1. Zirconium-oxide ceramic 3 from manufacturers:
 - Cercon, Degudent GmbH, Hanue-Wolfgang, Germany
 - Lava, 3M ESPE , Seefeld, Germany
 - Katana, Nuvodent, Noritake Dental Supply, Japan
2. Resin composite (Filtek Z250, 3M ESPE, St. Paul, MN, USA)
3. Resin cement 3 types
 - PanaviaF 2.0 ; Kuraray Dental, USA
 - Superbond C&B ; Sun Medical, Kyoto, Japan
 - RelyX™ Unicem; 3M ESPE, St. Paul, MN, USA

Other instruments and material used for test in this study

- Grinder-polisher Ecomet®250 (Buehler, IL, USA) (Figure 2)
- Sandblasting machine P-G400 (Harnisch&Rieth, Winterbach, Germany)
(Figure 3)
- Ultrasonic cleaner 5210 (Branson, Germany) (Figure 4)
- Light curing unit XL 3000 (3M ESPE, St. Paul, MN, USA) (Figure 5)
- Durameter stand (Pacific Tranducer Corp, Los Angeles, CA, USA) (Figure 6)

- High speed cutting machine (Model Isomet®4000 Linear precision saw, Buehler, IL, USA) (Figure 7)
- Veneer digital caliper (Mitutoyo, Japan) (Figure 8)
- Universal testing machine (SHIMADZU EZ S, 500 N) (Figure 9)
- Stereomicroscope (Olympus SZ-CTV, Olympus Co., Tokyo, Japan) (Figure 10)
- Scanning electron microscopy (SEM, XL 20; Philips, Eindhoven, The Netherlands) (Figure 11)
- Model Repair II Blue (Dentsply-Sankin, Ohtawara, Japan)
- Silicon carbide paper 100 220 400 800 1200 and 2000-grit (TOA,Sankyo, Thailand)
- 50µm aluminumoxide particles for sandblasting
- Polypropylene mold, size 5×5×10 mm.
- Mylar strip, TDV Dental Ltd., Santa Catarina, Brazil.



Figure 2 Ecomet®250 grinder-polisher



Figure 3 Sandblasting machine P-G400



Figure 4 Ultrasonic cleaner 5210



Figure 5 Light curing unit XL 3000



Figure 6 Durameter stand



Figure 7 High speed cutting machine



Figure 8 Veneer digital caliper



Figure 9 Universal testing machine
SHIMADZU EZ S



Figure 10 Stereomicroscope



Figure 11 Scanning electron microscope

A: Specimen preparation

- **Zirconium-oxide block**

Three fully sintered rectangular blocks (5x5x10 mm) (Figure12) of three brands of zirconium-oxide ceramics [Cercon(Degudent GmbH, Hanue-Wolfgang, Germany), Lava(3M ESPE , Seefeld, Germany), Katana (Nuvodent, Noritake Dental Supply, Japan)] were fabricated according to the manufacturer's instructions. Ceramic surfaces were finished with 100, 220, 400, 800, 1200 and 2000-grit silicon carbide abrasive (TOA, Sankyo, Thailand) in a polishing machine (Grinder-polisher Ecomet®250, Buehler, IL, USA) and sandblasted (Sandblasting machine, P-G400; Harnisch&Rieth, Winterbach, Germany) with 50µm aluminum-oxide particles at 0.4-0.5 MPa for 10 seconds at distance of 1 cm followed by ultrasonic cleaning in distilled water for 10 minutes.



Figure 12 Fully sintered rectangular zirconium-oxide ceramic blocks (5x5x10 mm)

- **Resin composite block**

Composite resin blocks (Filtek Z250, shade A2; 3M ESPE, St. Paul, MN, USA) with same size (5x5x10 mm) (Figure13) were prepared by mold made from silicon

impression material. Composite resin layers were incrementally (2mm) condensed into the mold to fill up the mold with a clean plastic filling instrument to avoid any contamination. Each layer was light polymerized for 40 seconds (Light curing unit XL 3000, 3M ESPE, St. Paul, MN, USA). The last increment of composite was covered with a Mylar strip and glass slide. The resin surfaces were finished with the same grit silicon carbide abrasive and cleaned for 10 minutes in an ultrasonic bath (Ultrasonic cleaner 5210, Branson, Germany).

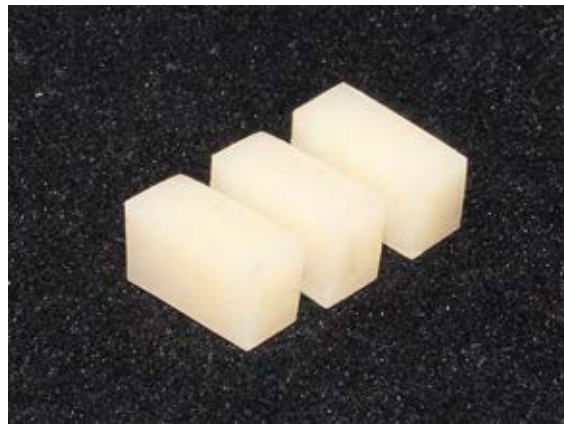


Figure 13 Composite resin blocks (5x5x10 mm)

B: Cementation

Table 5 Group division and amount of sample of each group.

Group	Specimen	No. of specimens
1	Cercon/ Panavia	10
2	Cercon/ Superbond	10
3	Cercon/ Unicem	10
4	Lava/ Panavia	10

5	Lava/ Superbond	10
6	Lava/ Unicem	10
7	Katana/ Panavia	10
8	Katana/ Superbond	10
9	Katana/ Unicem	10



Figure 14 PanaviaF 2.0; Kuraray Dental, USA



Figure 15 Superbond C&B; Sun Medical

Kyoto, Japan



Figure 16 RelyX™ Unicem; 3M ESPE, St. Paul, MN, USA

Before cementation, the zirconia block and composite resin block were rinsed thoroughly with water and dried. The cements were prepared and mixed, according to the manufacturers' instructions. Groups 1,4 and 7 (using PanaviaF 2.0): the equal amounts of paste A and paste B of PanaviaF 2.0 were dispensed and mixed sufficiently on a mixing pad for 20 seconds. The mixed paste was applied to the zirconia block surface which was seated on top of the composite resin block and loaded with 1kg (Durameter stand, Pacific Tranducer Corp, Los Angeles, CA, USA) for 60 seconds, then the excess cement was removed. The samples were light-cured for 20 seconds per surface with a dental curing light. Groups 2,5 and 8 (using Superbond C&B): the cement was mixed by dispensing the four drops of liquid into the chilled dispensing dish (10 - 16°C) and mixed with catalyst. Stirred lightly with a brush, then the polymer powder added to the Activated Liquid. Stirred lightly with a brush and applied immediately after mixing, using a brushing technique to the zirconia block surface which was seated on top of the composite resin block and 1kg load was applied for 8 minutes then the excess cement was removed with alcohol pellets. Groups 3,6 and 9 (using RelyX Unicem): the cement was mixed by inserting capsule into the activator for 2-4 seconds. The activated capsule was inserted into mixing device (triturator/amalgamator: 3M™ ESPE™ RotoMix™ Capsule Mixing Unit) and mixed for 10 seconds. The capsule was removed from mixing device and insertd into Applier, then nozzle opened and cement dispensed directly onto zirconia surface which was seated on top of the composite resin block and 1kg load was applied for 60 seconds and the excess cement was removed. Also, the samples were light-cured for 20 seconds per surface with a dental curing light.



Figure 17 The zirconia-resin specimens were bonded with cement.

Table 6 Composition of dental cements used in this study.

Material and composition	Lot No.
<p>PanaviaF 2.0 (Kuraray, Okayama, Japan)</p> <p>-PanaviaF 2.0 paste A: 10-Methacryloyloxydecyl dihydrogen phosphate, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated silica filler, Silanated colloidal silica, dl-Camphorquinone, Catalysts, Initiators, Others</p> <p>-PanaviaF 2.0 paste B: Sodium fluoride, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated barium glass filler, Catalysts, Accelerators, Pigments, Others</p>	051359
<p>Superbond C&B (Sun Medical, Kyoto, Japan)</p> <p>Polymer : PMMA</p> <p>Monomer: MMA, 4-META</p> <p>Catalyst S: TBB, acetone</p>	ER1

RelyX™ Unicem (3M ESPE, St. Paul, MN, USA)

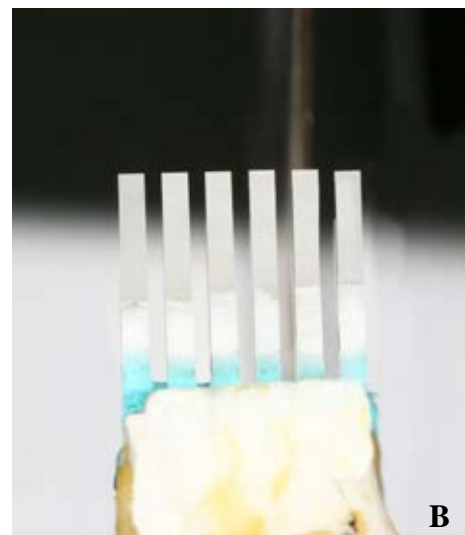
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Powder : Glass powder, silica sil, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator (light and self cure initiator).

Liquid : Methacrylated phosphoric ester, dimenthacrylates, acetate, stabilizer, initiator (light and self cure initiator).

C: Specimen cutting

The zirconia-resin specimens were bonded with Model Repair II Blue (Dentsply-Sankin, Ohtawara, Japan) to a metal base that was coupled to a cutting machine (High speed cutting machine, Model Isomet®4000 Linear precision saw, Buehler, IL, USA). Each bonded specimen was vertically sectioned under running water coolant into 1 mm thick slabs and further into beams with cross-sectioned areas of $1 \pm 0.1 \text{ mm}^2$



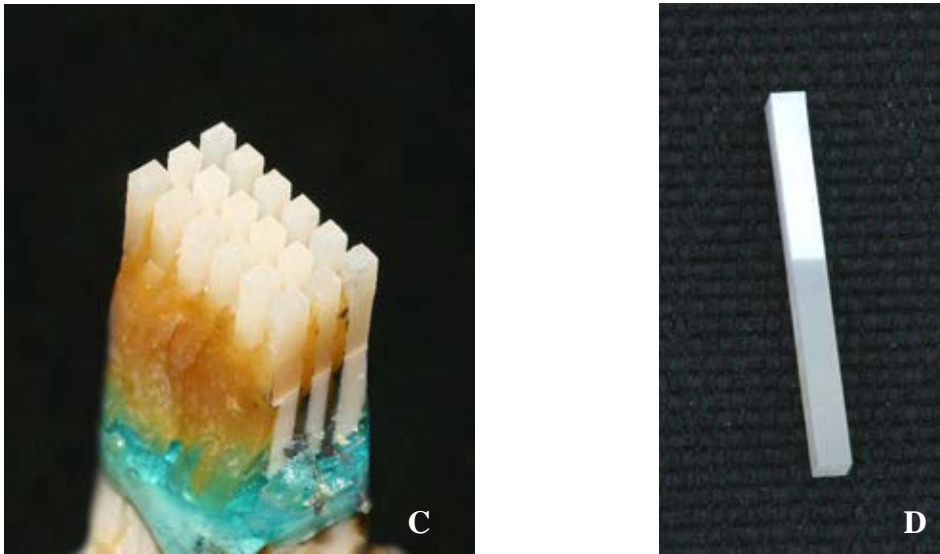


Figure 18 (A,B,C,D): The zirconia resin block is cut into microbars. Microbars with cross-sectioned areas of $1 \pm 0.1 \text{ mm}^2$ for microtensile bond strength test

D: Microtensile bond strength test

The dimensions of each specimen were measured with veneer digital caliper (Mitutoyo, Japan). The microbar specimens were attached to the flat grip with Model Repair II Blue. The MTBS (MPa) was measured by applying tensile load to the bonded interface using a universal testing machine SHIMADZU EZ S, 500 N at a crosshead speed of 0.5 mm/min.

E: Analysis of failure mode

The fractured specimens were evaluated under a stereomicroscope (Olympus SZ-CTV, Olympus Co., Tokyo, Japan) at 40x magnification to determine the mode of failure. Fracture patterns were classified as cohesive (within the cement or ceramic), adhesive (between the composite and the cement or at the cement/dentin level) or mixed (adhesive and cohesive fractures occurred simultaneously). Then the

fractured zirconium-oxide ceramics were examined under a scanning electron microscope (SEM, XL 20; Philips, Eindhoven, The Netherlands).

F: Statistical analysis

Statistical analysis was performed using two-way analysis of variance (ANOVA) to study the contributions of the resin cement types, the brands of zirconium-oxide ceramic and their interaction on MTBS. Multiple comparisons were conducted using Tukey's tests at p -value = 0.05. Statistical analysis was carried out using computer software (SigmaStat Version 17.0, SPSS, Inc., Chicago, USA).

CHAPTER IV

RESULTS

4.1 Microtensile bond strength analysis.

The mean and standard deviation of microtensile bond strength values of experiment groups are reported in Table 7.

Table 7 The mean and standard deviation of MTBS of the experiment groups.

Group	Specimen	Mean \pm SD
1	Cercon/ Panavia	43.28 \pm 5.63
2	Cercon/ Superbond	45.40 \pm 7.14
3	Cercon/ Unicem	51.60 \pm 6.86
4	Lava/ Panavia	47.86 \pm 3.66
5	Lava/ Superbond	53.88 \pm 10.82
6	Lava/ Unicem	52.38 \pm 7.15
7	Katana/ Panavia	48.66 \pm 10.60
8	Katana/ Superbond	53.18 \pm 2.10
9	Katana/ Unicem	46.71 \pm 4.15

Table 8 Tests of Between-Subjects Effects, Dependent Variable:MTS.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1113.231a	8	139.154	2.814	.008
Intercept	217992.832	1	217992.832	4407.776	.000
Cement	313.056	2	156.528	3.165	.047
Zirconia	323.481	2	161.741	3.270	.043
Cement * Zirconia	476.694	4	119.173	2.410	.056
Error	4005.970	81	49.456		
Total	223112.034	90			
Corrected Total	5119.201	89			

a: R Squared = .217 (Adjusted R Squared = .140)

The MTBS of Lava/Superbond group (53.87 N/mm²) produced the highest MTBS, while Cercon/Panavia group (43.28 N/mm²) produced the lowest MTBS (Table 7). The two-way ANOVA and Tukey's post hoc test revealed that type of resin cement (F=3.165, P=0.047) and the brand of zirconium-oxide ceramic (F=3.270, P=0.043) significantly affected MTBS, while the interaction between brands of zirconium-oxide ceramic and types of resin cement (F=2.410, P=0.056) did not affect MTBS (Table 8).

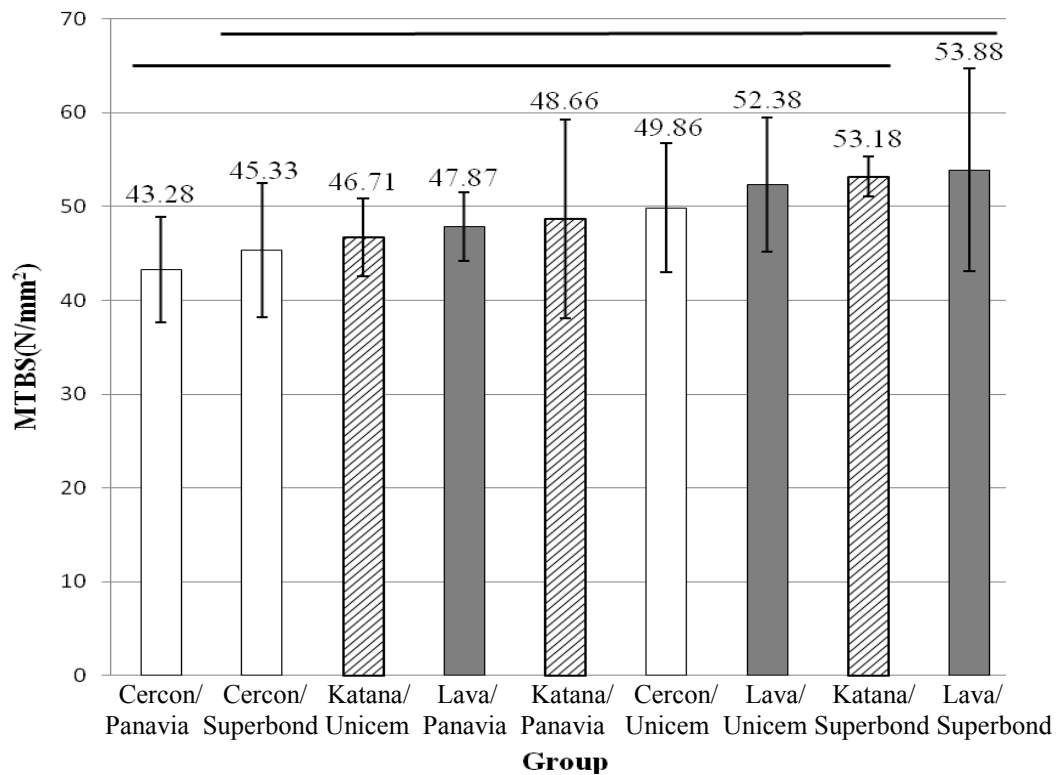


Figure 19 Mean and SD of MTBS values of experiment groups. — means no significant difference ($P > 0.05$).

The two-way ANOVA and Tukey's post hoc test revealed that type of cement has effect on MTBS as RelyX Unicem had significantly higher MTBS than PanaviaF 2.0 in Cercon, while not significant in Katana and Lava. Also, type of zirconium-oxide ceramic has effect on MTBS as three types of zirconium-oxide ceramic had no significant difference, when bonded with PanaviaF 2.0 and RelyX Unicem, while Lava had significantly higher MTBS than Cercon when bonded with Superbond (Figure19).

4.2 SEM observation of the ceramic surface.

SEM analysis at 2000x magnification, complementary to MTBS tests, revealed that PanaviaF 2.0 and RelyX Unicem demonstrated predominantly cohesive failure in resin cement as the surface of zirconia was covered by layer of resin cement. On both zirconia and resin composite site, Superbond C&B showed mix of adhesive and cohesive failure are on debonded surfaces. No adhesive failure was observed along the ceramic-cement interface or the resin composite-cement interface in all groups. The different percentages in pattern of failure are shown in Table 9.

Table 9 Failure types in each group of test groups.

Resin cement	Brand of zirconium-oxide ceramic	MTBS
PanaviaF 2.0	Cercon	100% Cohesive failure in resin cement
	Lava	100% Cohesive failure in resin cement
	Katana	100% Cohesive failure in resin cement
Superbond C&B	Cercon	70% Cohesive failure in resin cement+30% Mixed failure*
	Lava	70% Mixed failure*+ 30% Cohesive failure in resin cement
	Katana	60% Mixed failure*+ 40% Cohesive failure in resin cement
RelyX Unicem	Cercon	100% Cohesive failure in resin cement
	Lava	100% Cohesive failure in resin cement
	Katana	100% Cohesive failure in resin cement

*Mixed failure= Cohesive failure+ Adhesive failure.

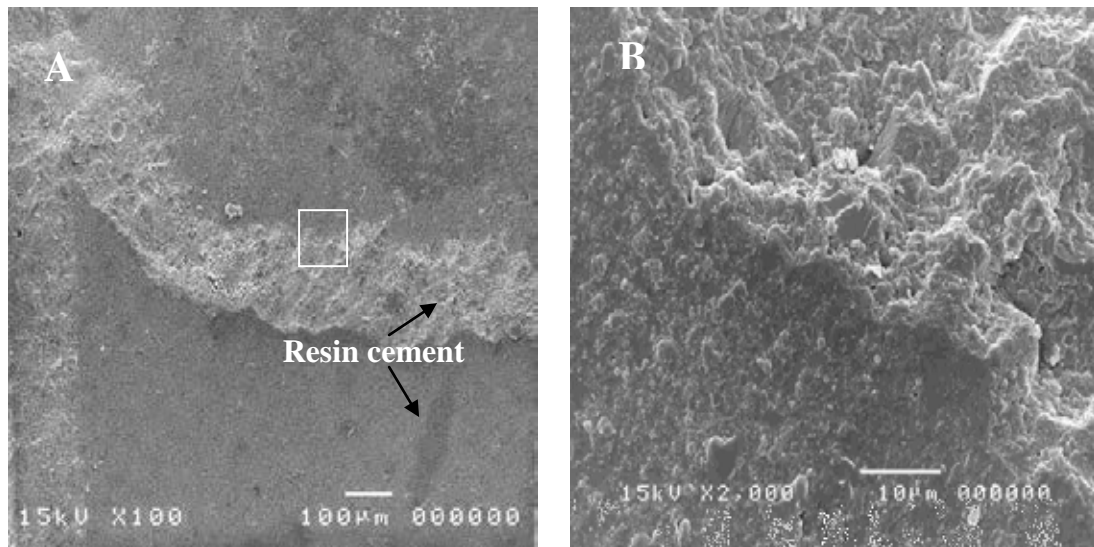


Figure 20 A: SEM image, 100 \times , demonstrating cohesive failure in resin cement observed with Cercon/Panavia **B:** SEM image, 2000 \times , demonstrating cohesive failure at the area white rectangular in A.

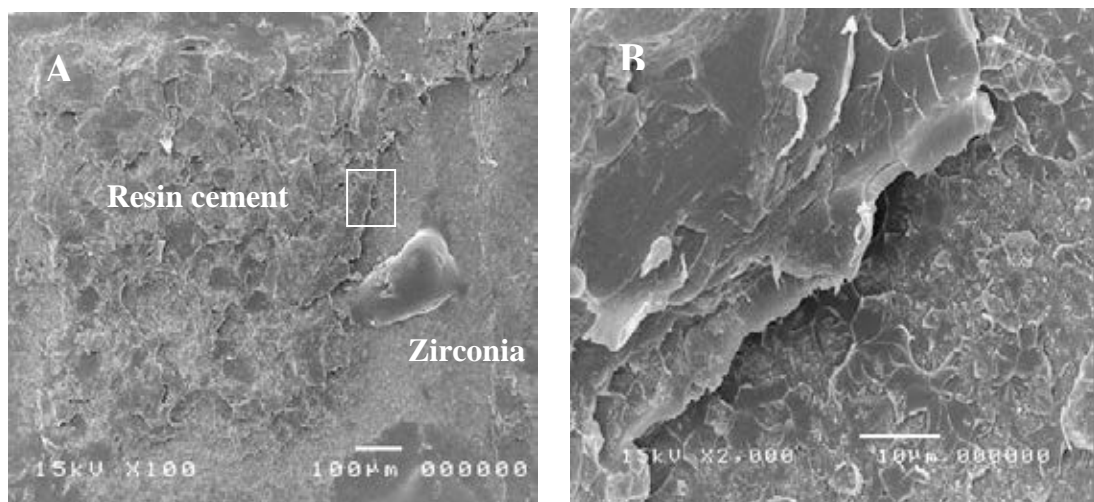


Figure 21 A: SEM image, 100 \times , demonstrating cohesive failure in resin cement observed with Cercon/Superbond **B:** SEM image, 2000 \times , demonstrating cohesive failure at the area white rectangular in A.

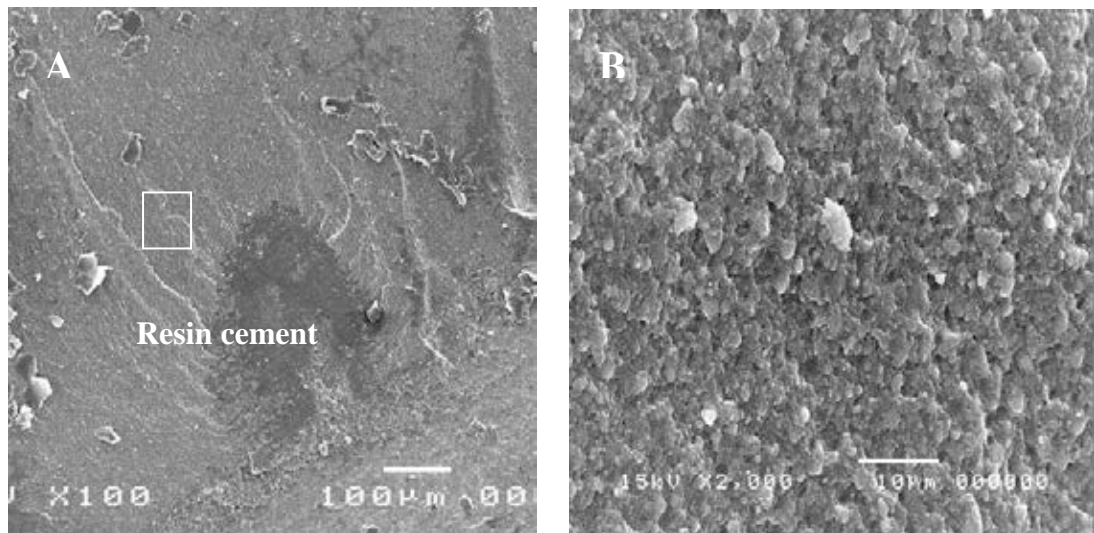


Figure 22 A: SEM image, 100 \times , demonstrating cohesive failure in resin cement observed with Cercon/Unicem **B:** SEM image, 2000 \times , demonstrating cohesive failure at the area white rectangular in A.

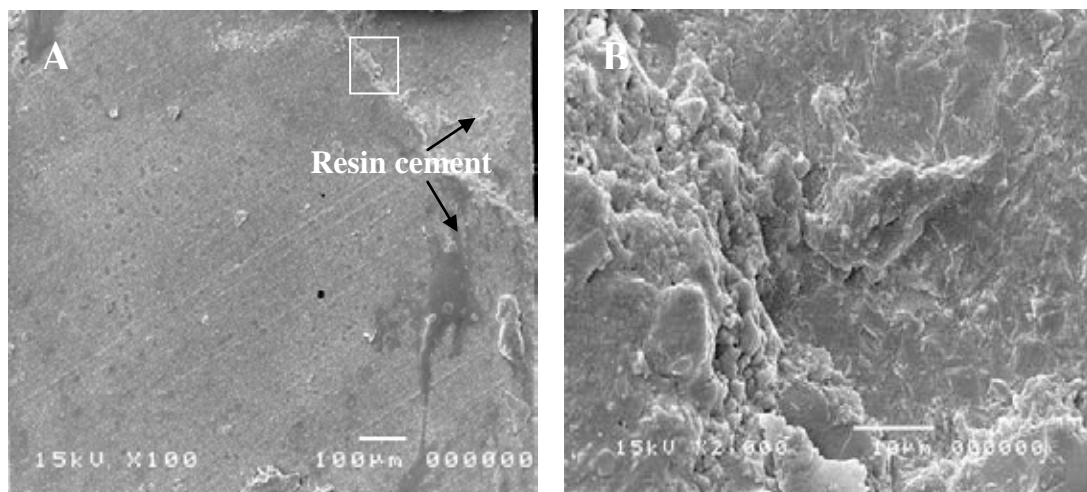


Figure 23 A: SEM image, 100 \times , demonstrating cohesive failure in resin cement observed with Lava/Panavia **B:** SEM image, 2000 \times , demonstrating cohesive failure at the area white rectangular in A.

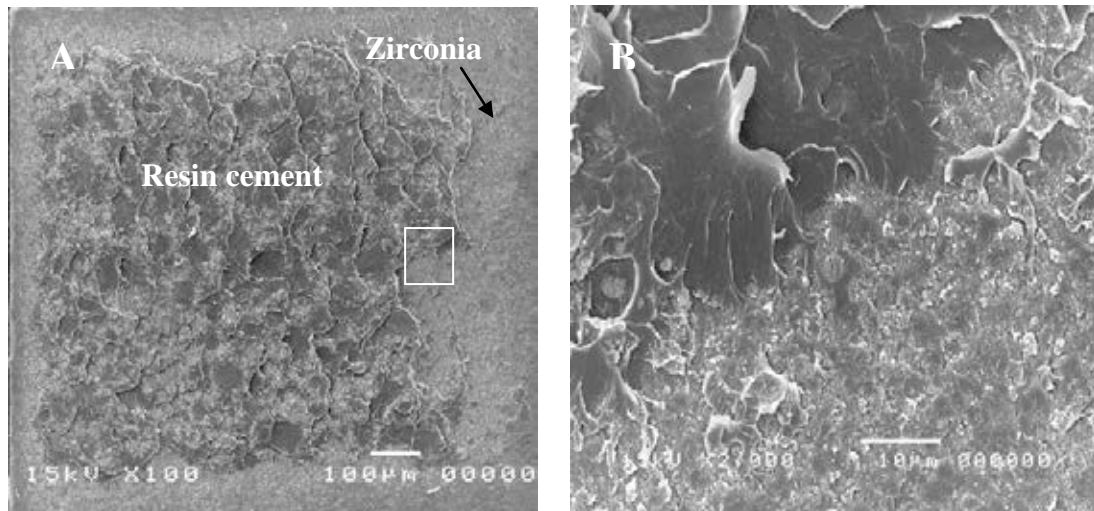


Figure 24 A: SEM image, 100 \times , demonstrating cohesive failure in resin cement observed with Lava/Superbond **B:** SEM image, 2000 \times , demonstrating cohesive failure at the area white rectangular in A.

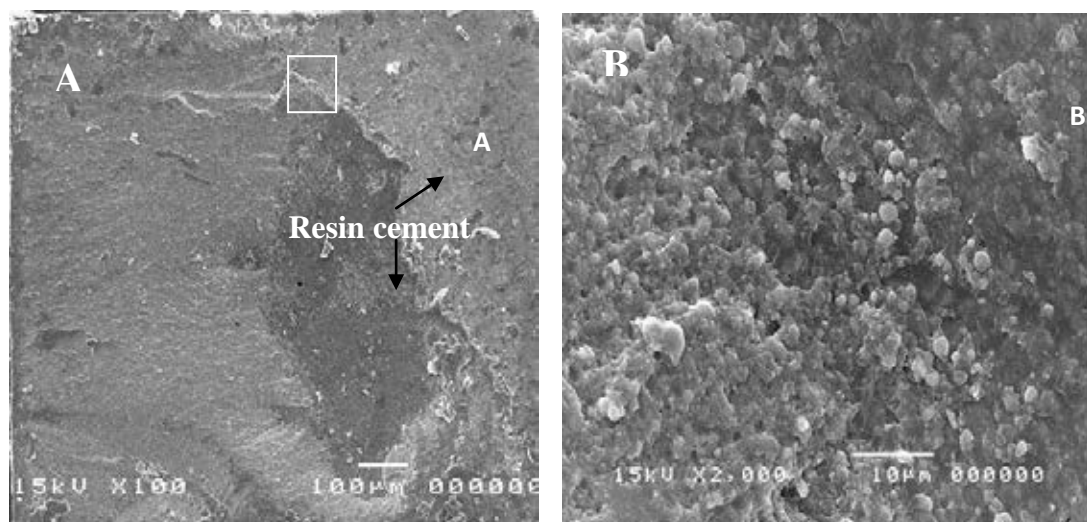


Figure 25 A: SEM image, 100 \times , demonstrating cohesive failure in resin cement observed with Lava/Unicem **B:** SEM image, 2000 \times , demonstrating cohesive failure at the area white rectangular in A.

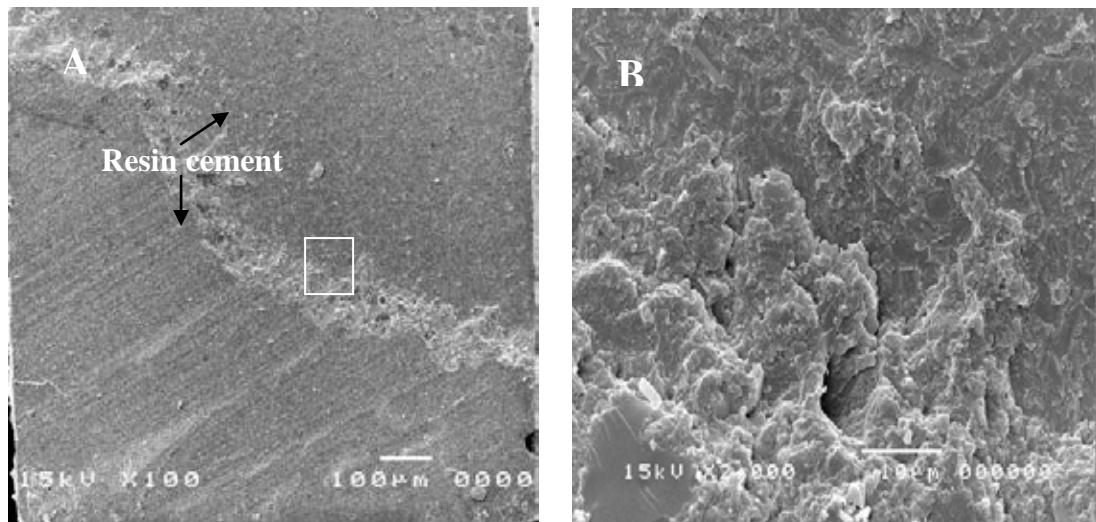


Figure 26 A: SEM image, 100×, demonstrating cohesive failure in resin cement observed with Katana/Panavia **B:** SEM image, 2000×, demonstrating cohesive failure at the area white rectangular in A.

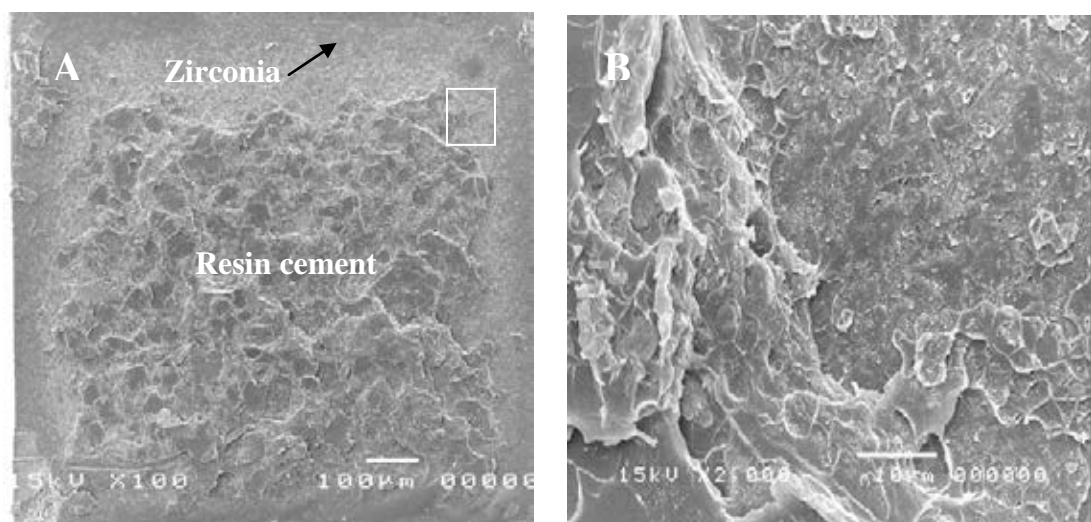


Figure 27 A: SEM image, 100×, demonstrating cohesive failure in resin cement observed with Katana/Superbond **B:** SEM image, 2000×, demonstrating cohesive failure at the area white rectangular in A.

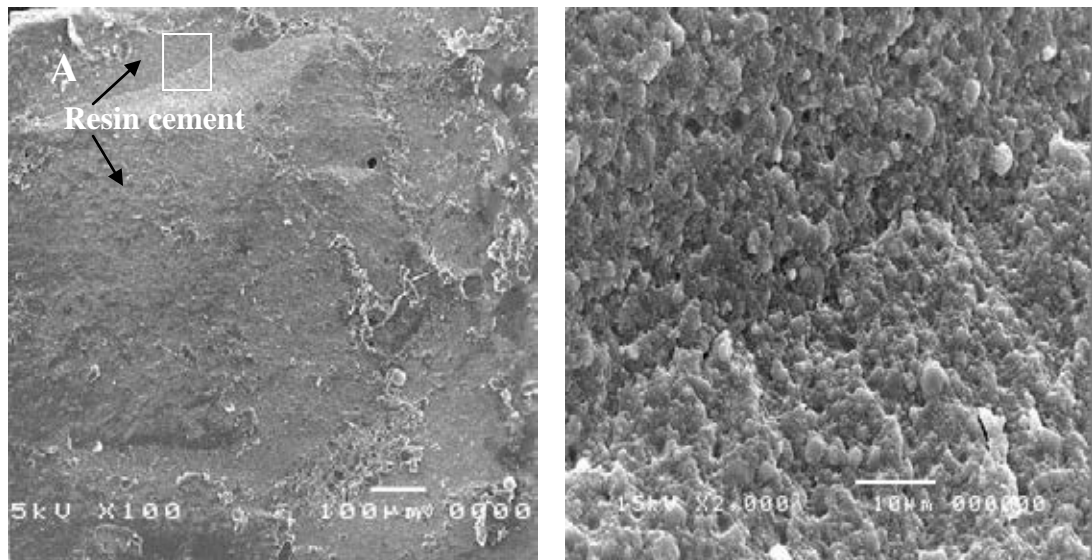


Figure 28 A: SEM image, 100×, demonstrating cohesive failure in resin cement observed with Katana/Unicem **B:** SEM image, 2000×, demonstrating cohesive failure at the area white rectangular in A.

CHAPTER V

DISCUSSION

The results of this study showed that MTBS between zirconia and resin cement depends on types of resin cement and brands of zirconium-oxide ceramic. While the interaction between zirconium-oxide ceramic and types of resin cement did not affect the value of MTBS (Table 8), two-way ANOVA revealed that type of zirconium-oxide ceramic has effect on MTBS as Lava had significantly higher MTBS than Cercon when bonded with Superbond, while three types of zirconium-oxide ceramic had no significant difference, when bonded with PanaviaF 2.0 and RelyX Unicem. This might be because each brand of zirconium-oxide ceramic might have differences in many aspects (Table 11), such as powder properties: composition, impurities, particle size, crystalline size and etc., which affect the final properties of the zirconia restoration. The composition and impurities affect the translucency, strength, and density of the final zirconia body, while the particle size and crystalline size affect the sintering process, the sintering time and final mechanical and physical properties. Three brands of zirconium-oxide ceramic in this study consist of yttrium oxide in the range of 3 - 5.2% to treat tetragonal zirconia polycrystals. Data from instruction revealed that Cercon also treated zirconium-oxide with a very small concentration of alumina (0.2 - 0.5%) to prevent leaching of the yttrium oxide. This combination ensures the safety and longevity of zirconia restorations. Besides, the grain size or consistency of the powder has an effect on

bonding strength. Variations in grain size distribution (Cercon 0.5 μ m and Lava 0.07-0.3 μ m) affect results in porosity, surface roughness and value of MTBS [62].

Table 10 Differences of each brand of zirconium-oxide ceramic [61-63].

	Cercon	Lava	Katana
%Zirconium oxide	92.3%	97%	94.4%
% Yttrium oxide	5%	3%	5.4%
Binder	Hafnium oxide < 2%	No	No
Grain size of powder	0.5 μ m	0.07-0.3 μ m	N/A
Sintering technique	Non-HIP (Pre-sintered)	Non-HIP (Pre-sintered)	Non-HIP (Non-pre-sintered)
Final sintering temperature	> 1,350°C	1,360°C–1,530°C	1,400°C -1,500°C
Time	7 hours	8 hours	11 hours

Furthermore, other major variables include the pressing, debinding and pre-sintering of the powder to produce blocks. Proper processing and pre-sintering are critical to achieve the required shrinkage and density. If the powders are not correctly pressed, a restoration can exhibit lower densities and internal voids, which can cause fractures, and also result in non-homogeneous shrinkage, which can warp longer units, such as bridges. The porosity decreasing could improve mechanical properties of the specimens and thus increasing the density and interfacial bonding strength.

Moreover, the most important processing factor is the firing of the pre-sintered restoration process, which produces a homogeneous sub-micron crystal structure that is needed for the transformation toughening to be present, and gives the restorations their very high strength. It is critical that firing parameters are followed precisely. An optimized temperature to generate suitable strength and millability, which influences the homogeneity and the density distribution of the material and coloring of the zirconia by immersing frameworks in a dyeing liquid, might also lead to differences in the bonding surface of zirconia. Finally, it is notable that percent of zirconium-oxide in Lava(97%) is more than in Katana(94.4%) and Cercon(92.3%) (Table10), it might affect to MTBS due to hydroxyl groups that present on the zirconia surface, which created a bond between resin cement and zirconium-oxide ceramic because the surface of zirconium-oxide ceramic is coated with a passive film of zirconium oxide.

Two-way ANOVA revealed that type of cement has effect on MTBS as RelyX Unicem had significantly higher MTBS than PanaviaF 2.0 in Cercon, while not significant in Katana and Lava. This could be because RelyX Unicem is filled with reactive glass 72% weight which less than in Panavia F 2.0, is filled with reactive glass 78% weight [64]. It might affect the fluidity of cement and film thickness, film thickness of RelyX Unicem might be thinner than PanaviaF 2.0. In the current International Organization for Standardization standards, a film thickness is required at the time of seating of no greater than 50 μ m for resin-based cements [65]. Hibino (1990) revealed that greater resin cement film thickness resulted in significantly decreased bond strength [66]. Therefore, RelyX Unicem might have higher MTBS than PanaviaF 2.0.

SEM observed in this study revealed that Panavia F 2.0 and RelyX Unicem demonstrated predominantly cohesive failure in resin cement as the surface of zirconia was covered by layer of resin cement. Superbond C&B showed mix of adhesive and cohesive failure on debonded surfaces on both zirconia and resin composite sites. No complete adhesive failure was observed along the ceramic-cement interface or the resin composite-cement interface in all groups. Cekic-Nagas et al. (2010) showed that the fracture modes in groups with a cement thickness of 50 μ m were predominantly adhesive between the resin cement and ceramic. However, the failures were mostly cohesive in the resin cement in the groups with a resin cement thickness of 100 μ m [67]. The thicker cement film thickness could be related to a reduced degree of conversion and, eventually, incomplete polymerization of the thicker cement layer, which might induce the cement layer to be weak and to be a cause of cohesive failure in resin cement. The other reason might be that 10-methacryloyloxydecyl dihydrogenphosphate (MDP) is presented in Panavia F 2.0 and RelyX Unicem is contained phosphoric acid monomer and methacrylate monomers, where the phosphate ester group of these cements has a chemical bond to the zirconium-oxides. Therefore, it might be successful in establishing good bond strength with zirconia. The performance of this bond should affect mode of failure on both Panavia F 2.0 and RelyX Unicem, which showed cohesive failure in resin cement. Careful interpretation of the failure mode is required to prevent inappropriate conclusions. Nevertheless, this study had controlled luting procedures by loaded with a constant load of 1 kg (98N) [31,50,68-69], did not control the film thickness of resin cement. Further study should be carried out to control film thickness by cover block with masking tapes to standardize cement thickness.

MTBS is a mechanical test that permits a more homogeneous distribution of stress on specimens. The advantages of the MTBS test, such as the nominal bond strength values being higher than other tests that utilize a larger interface (a tensile load is applied to a very small cross-section of the bonded interface between test substrates and the adhesive material of interest), involve a large number of microbars per specimen and promote ease of analysis of fracture surface. Sano et al. (1994) showed that the MTBS values are inversely related to the bonded surface area [53-55] and that although much higher bond strength values were measured, most failures still occurred at the interface. The reason for higher bond strength value might be a small adhesive interface area contains fewer defects compared to larger specimen interface area. Fewer defects would reduce variations and increase MTBS value. However, the MTBS testing is rather labor-intensive, technically demanding, and requires careful handling of the fragile specimens. Furthermore, the samples, which are so small, can dehydrate rapidly. El Zohairy et al. (2003) showed that the size and method of attachment has an influence on the observed bond strength [70]. Aboushelib et al. (2005) revealed that the highest area of stress concentration in MTBS was located away from the bonded interface, which can play a role in case of relatively stiff or weak materials. Moreover, it was shown that stress distribution in the microbars were not homogeneous, probably depending on the way specimens were attached to the device [71]. All of the disadvantages could also affect the MTBS value. So this study has taken special care to reduce the production of microfractures at the interface during specimen preparation which might weaken the bond and reduce the actual value. Also, we have been aware of the way specimens should be parallel to the joint of the device. The original specimen design was dumbbell or

hourglass shaped and a non-trimming version has been evolved from this design. The non-trimming design is easier to prepare with a more precise bonded interface, less labor-intensive and produces less stress, heat and premature failure on the bonded interface during specimen preparation. In this study we obtained rectangular sticks of about $1 \pm 0.1 \text{ mm}^2$ bonding surface area, where, due to a small surface area, we expected the stress distribution to be uniform, and the MTBS test measurements to truly express the interfacial bond strength.

The outcome of this study indicated that relatively recent MTBS was not based on the combination of type of resin cement and brands of zirconium-oxide ceramics. Also, it could choose any type of resin cement in combination with any brand of zirconium-oxide ceramics, it would provide the same results. However, the results of this experiment provide only three types of resin cements to three brands of zirconium-oxide ceramics and due to the high SD of some groups in the present investigation, a greater number of specimens per group might provide different results. Other limitation of this study might be the lack of thermocycling, although there are controversial reports on the effect of thermocycling in the literature [22]. It can be projected that long-term thermocycling and water storage may significantly affect the bond strength of resin-based luting agents to zirconia [34]. Clinical trials are needed to refine these conclusions because the cement–ceramic adhesion is susceptible to thermal, chemical and mechanical influences under intraoral conditions, which will be the focus of future studies.

CHAPTER VI

CONCLUSIONS

The study has been performed to test the bonding between three types of resin cement and three brands of zirconium-oxide ceramic by using MTBS. Within the limits of this study, the conclusions were drawn:

1. Type of resin cement has effect on MTBS when bonded with zirconium-oxide ceramic($P=0.047$).
2. Type of zirconium-oxide ceramic has effect on MTBS when bonded with resin cement($P=0.043$).
3. The MTBS was not based on the combination of type of resin cement and brands of zirconium-oxide ceramics($P=0.056$).

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APPENDIX

One-Sample Kolmogorov-Smirnov Test

group		MTS	
panavia+katana	N	10	
	Normal Parameters ^{a,b}	Mean	48.6564
		Std. Deviation	10.59953
	Most Extreme Differences	Absolute	.205
		Positive	.137
		Negative	-.205
	Kolmogorov-Smirnov Z	.648	
	Asymp. Sig. (2-tailed)	.795	
panavia+lava	N	10	
	Normal Parameters ^{a,b}	Mean	47.8648
		Std. Deviation	3.65740
	Most Extreme Differences	Absolute	.150
		Positive	.122
		Negative	-.150
	Kolmogorov-Smirnov Z	.475	
	Asymp. Sig. (2-tailed)	.978	
panavia+cercon	N	10	
	Normal Parameters ^{a,b}	Mean	43.2772
		Std. Deviation	5.62484
	Most Extreme Differences	Absolute	.279
		Positive	.279
		Negative	-.128
	Kolmogorov-Smirnov Z	.882	
	Asymp. Sig. (2-tailed)	.417	
superbond+katana	N	10	
	Normal Parameters ^{a,b}	Mean	53.1746
		Std. Deviation	2.09958
	Most Extreme Differences	Absolute	.170
		Positive	.170
		Negative	-.122
Kolmogorov-Smirnov Z	.537		

		Asymp. Sig. (2-tailed)	.935
superbond+lava	N		10
	Normal Parameters ^{a,b}	Mean	53.8745
		Std. Deviation	10.81563
	Most Extreme Differences	Absolute	.177
		Positive	.144
		Negative	-.177
	Kolmogorov-Smirnov Z		.560
	Asymp. Sig. (2-tailed)		.912
superbond+cercon	N		10
	Normal Parameters ^{a,b}	Mean	45.3986
		Std. Deviation	7.13948
	Most Extreme Differences	Absolute	.160
		Positive	.119
		Negative	-.160
	Kolmogorov-Smirnov Z		.507
	Asymp. Sig. (2-tailed)		.959
unicem+katana	N		10
	Normal Parameters ^{a,b}	Mean	46.7112
		Std. Deviation	4.15177
	Most Extreme Differences	Absolute	.255
		Positive	.124
		Negative	-.255
	Kolmogorov-Smirnov Z		.805
	Asymp. Sig. (2-tailed)		.536
unicem+lava	N		10
	Normal Parameters ^{a,b}	Mean	52.3807
		Std. Deviation	7.14548
	Most Extreme Differences	Absolute	.203
		Positive	.093
		Negative	-.203
	Kolmogorov-Smirnov Z		.641
	Asymp. Sig. (2-tailed)		.805
unicem+cercon	N		10

Normal Parameters ^{a,b}		Mean	51.5994
		Std. Deviation	6.86214
Most Extreme Differences		Absolute	.213
		Positive	.213
		Negative	-.138
Kolmogorov-Smirnov Z			.674
Asymp. Sig. (2-tailed)			.755

a. Test distribution is Normal.

b. Calculated from data.

Univariate Analysis of Variance

Between-Subjects Factors

		Value Label	N
Cement	1	panavia	30
	2	superbond	30
	3	Unicem	30
Zirconia	1	katana	30
	2	lava	30
	3	cercon	30

Tests of Between-Subjects Effects

Dependent Variable: MTS

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1113.231 ^a	8	139.154	2.814	.008
Intercept	217992.832	1	217992.832	4407.776	.000
Cement	313.056	2	156.528	3.165	.047
Zirconia	323.481	2	161.741	3.270	.043
Cement * Zirconia	476.694	4	119.173	2.410	.056
Error	4005.970	81	49.456		
Total	223112.034	90			
Corrected Total	5119.201	89			

a. R Squared = .217 (Adjusted R Squared = .140)

Post Hoc Tests

Multiple Comparisons

MTS

Tukey HSD

(I) group	(J) group	Mean Difference (I- J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
panavia+katana	panavia+lava	.79160	3.14504	1.000	-9.2323	10.8155
	panavia+cercon	5.37919	3.14504	.738	-4.6447	15.4031
	superbond+katana	-4.51822	3.14504	.880	-14.5421	5.5057
	superbond+lava	-5.21814	3.14504	.769	-15.2420	4.8058
	superbond+cercon	3.25774	3.14504	.981	-6.7662	13.2816
	unicem+katana	1.94515	3.14504	.999	-8.0787	11.9690
	unicem+lava	-3.72436	3.14504	.958	-13.7483	6.2995
	unicem+cercon	-2.94304	3.14504	.990	-12.9669	7.0809
panavia+lava	panavia+katana	-.79160	3.14504	1.000	-10.8155	9.2323
	panavia+cercon	4.58759	3.14504	.871	-5.4363	14.6115
	superbond+katana	-5.30982	3.14504	.752	-15.3337	4.7141
	superbond+lava	-6.00974	3.14504	.608	-16.0336	4.0142
	superbond+cercon	2.46614	3.14504	.997	-7.5578	12.4900
	unicem+katana	1.15355	3.14504	1.000	-8.8703	11.1774
	unicem+lava	-4.51596	3.14504	.880	-14.5399	5.5079
	unicem+cercon	-3.73464	3.14504	.957	-13.7585	6.2893
panavia+cercon	panavia+katana	-5.37919	3.14504	.738	-15.4031	4.6447
	panavia+lava	-4.58759	3.14504	.871	-14.6115	5.4363
	superbond+katana	-9.89741	3.14504	.056	-19.9213	.1265
	superbond+lava	-10.59733	3.14504	.030	-20.6212	-.5734
	superbond+cercon	-2.12145	3.14504	.999	-12.1453	7.9024
	unicem+katana	-3.43404	3.14504	.974	-13.4579	6.5899
	unicem+lava	-9.10355	3.14504	.105	-19.1274	.9203
	unicem+cercon	-8.32223	3.14504	.184	-18.3461	1.7017
superbond+katana	panavia+katana	4.51822	3.14504	.880	-5.5057	14.5421
	panavia+lava	5.30982	3.14504	.752	-4.7141	15.3337
	panavia+cercon	9.89741	3.14504	.056	-.1265	19.9213

	superbond+lava	-69992	3.14504	1.000	-10.7238	9.3240
	superbond+cercon	7.77596	3.14504	.261	-2.2479	17.7999
	unicem+katana	6.46337	3.14504	.511	-3.5605	16.4873
	unicem+lava	.79386	3.14504	1.000	-9.2300	10.8178
	unicem+cercon	1.57518	3.14504	1.000	-8.4487	11.5991
superbond+lava	panavia+katana	5.21814	3.14504	.769	-4.8058	15.2420
	panavia+lava	6.00974	3.14504	.608	-4.0142	16.0336
	panavia+cercon	10.59733	3.14504	.030	.5734	20.6212
	superbond+katana	.69992	3.14504	1.000	-9.3240	10.7238
	superbond+cercon	8.47588	3.14504	.166	-1.5480	18.4998
	unicem+katana	7.16329	3.14504	.368	-2.8606	17.1872
	unicem+lava	1.49378	3.14504	1.000	-8.5301	11.5177
	unicem+cercon	2.27510	3.14504	.998	-7.7488	12.2990
superbond+cercon	panavia+katana	-3.25774	3.14504	.981	-13.2816	6.7662
	panavia+lava	-2.46614	3.14504	.997	-12.4900	7.5578
	panavia+cercon	2.12145	3.14504	.999	-7.9024	12.1453
	superbond+katana	-7.77596	3.14504	.261	-17.7999	2.2479
	superbond+lava	-8.47588	3.14504	.166	-18.4998	1.5480
	unicem+katana	-1.31259	3.14504	1.000	-11.3365	8.7113
	unicem+lava	-6.98210	3.14504	.403	-17.0060	3.0418
	unicem+cercon	-6.20078	3.14504	.567	-16.2247	3.8231
unicem+katana	panavia+katana	-1.94515	3.14504	.999	-11.9690	8.0787
	panavia+lava	-1.15355	3.14504	1.000	-11.1774	8.8703
	panavia+cercon	3.43404	3.14504	.974	-6.5899	13.4579
	superbond+katana	-6.46337	3.14504	.511	-16.4873	3.5605
	superbond+lava	-7.16329	3.14504	.368	-17.1872	2.8606
	superbond+cercon	1.31259	3.14504	1.000	-8.7113	11.3365
	unicem+lava	-5.66951	3.14504	.680	-15.6934	4.3544
	unicem+cercon	-4.88819	3.14504	.826	-14.9121	5.1357
unicem+lava	panavia+katana	3.72436	3.14504	.958	-6.2995	13.7483
	panavia+lava	4.51596	3.14504	.880	-5.5079	14.5399
	panavia+cercon	9.10355	3.14504	.105	-.9203	19.1274
	superbond+katana	-.79386	3.14504	1.000	-10.8178	9.2300
	superbond+lava	-1.49378	3.14504	1.000	-11.5177	8.5301

	superbond+cercon	6.98210	3.14504	.403	-3.0418	17.0060
	unicem+katana	5.66951	3.14504	.680	-4.3544	15.6934
	unicem+cercon	.78132	3.14504	1.000	-9.2426	10.8052
unicem+cercon	panavia+katana	2.94304	3.14504	.990	-7.0809	12.9669
	panavia+lava	3.73464	3.14504	.957	-6.2893	13.7585
	panavia+cercon	8.32223	3.14504	.184	-1.7017	18.3461
	superbond+katana	-1.57518	3.14504	1.000	-11.5991	8.4487
	superbond+lava	-2.27510	3.14504	.998	-12.2990	7.7488
	superbond+cercon	6.20078	3.14504	.567	-3.8231	16.2247
	unicem+katana	4.88819	3.14504	.826	-5.1357	14.9121
	unicem+lava	-.78132	3.14504	1.000	-10.8052	9.2426

*. The mean difference is significant at the 0.05 level.

Homogeneous Subsets

MTS

Tukey HSD^a

group	N	Subset for alpha = 0.05	
		1	2
panavia+cercon	10	43.2772	
superbond+cercon	10	45.3986	45.3986
unicem+katana	10	46.7112	46.7112
panavia+lava	10	47.8648	47.8648
panavia+katana	10	48.6564	48.6564
unicem+cercon	10	51.5994	51.5994
unicem+lava	10	52.3807	52.3807
superbond+katana	10	53.1746	53.1746
superbond+lava	10		53.8745
Sig.		.056	.166

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.

One way Panavia**ANOVA**

MTS

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	168.694	2	84.347	1.608	.219
Within Groups	1416.288	27	52.455		
Total	1584.982	29			

One way Superbond**ANOVA**

MTS

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	442.653	2	221.327	3.852	.034
Within Groups	1551.225	27	57.453		
Total	1993.878	29			

Multiple Comparisons

MTS

Tukey HSD

(I)	(J)	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Zirconia	Zirconia					
katana	lava	-.69992	3.38977	.977	-9.1046	7.7047
	cercon	7.77596	3.38977	.074	-.6287	16.1806
lava	katana	.69992	3.38977	.977	-7.7047	9.1046
	cercon	8.47588*	3.38977	.048	.0712	16.8805
cercon	katana	-7.77596	3.38977	.074	-16.1806	.6287
	lava	-8.47588*	3.38977	.048	-16.8805	-.0712

*. The mean difference is significant at the 0.05 level.

MTSTukey HSD^a

Zirconia	N	Subset for alpha = 0.05	
		1	2
cercon	10	45.3986	
katana	10	53.1746	53.1746
lava	10		53.8745
Sig.		.074	.977

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.

One way Unicem**ANOVA**

MTS

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	188.827	2	94.414	2.455	.105
Within Groups	1038.457	27	38.461		
Total	1227.285	29			

One way Cercon**ANOVA**

MTS

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	374.032	2	187.016	4.326	.023
Within Groups	1167.300	27	43.233		
Total	1541.332	29			

Multiple Comparisons

MTS

Tukey HSD

(I) Cement	(J) Cement	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
panavia	superbond	-2.12145	2.94052	.753	-9.4122	5.1693
	Unicem	-8.32223*	2.94052	.023	-15.6130	-1.0314
superbond	panavia	2.12145	2.94052	.753	-5.1693	9.4122
	Unicem	-6.20078	2.94052	.107	-13.4916	1.0900
Unicem	panavia	8.32223*	2.94052	.023	1.0314	15.6130
	superbond	6.20078	2.94052	.107	-1.0900	13.4916

*. The mean difference is significant at the 0.05 level.

MTS

Tukey HSD^a

Cement	N	Subset for alpha = 0.05	
		1	2
panavia	10	43.2772	
superbond	10	45.3986	45.3986
Unicem	10		51.5994
Sig.		.753	.107

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.

One way Lava**ANOVA**

MTS

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	195.807	2	97.904	1.619	.217
Within Groups	1632.711	27	60.471		
Total	1828.518	29			

One way Katana**ANOVA**

MTS

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	219.910	2	109.955	2.462	.104
Within Groups	1205.959	27	44.665		
Total	1425.869	29			

The microtensile bond strength values of each experiment groups									
MTS(N/mm²)	Cercon			Lava			Katana		
	Panavia	Superbond	unicem	Panavia	Superbond	unicem	Panavia	Superbond	unicem
1	43.41	48.54	44.45	52.69	42.92	49.30	32.45	53.24	47.37
2	53.80	40.54	49.35	48.03	56.22	58.59	45.70	53.85	49.06
3	43.57	55.54	54.94	46.46	67.52	53.35	53.57	51.35	47.97
4	43.05	37.82	52.20	50.34	56.54	48.69	55.44	56.15	47.28
5	42.80	48.35	54.70	50.55	66.43	50.49	56.76	56.30	50.62
6	37.43	48.75	42.41	45.96	50.45	36.26	40.76	50.53	43.21
7	41.87	53.34	52.78	48.56	66.43	51.89	60.88	51.23	51.50
8	51.49	42.94	49.14	51.60	41.69	57.89	58.07	51.62	37.03
9	35.75	32.15	48.73	41.79	37.83	55.23	51.48	52.42	47.77
10	39.61	46.02	67.30	42.68	52.72	62.12	31.46	55.05	45.30
Average	43.28	45.40	51.60	47.86	53.87	52.38	48.66	53.17	46.71
SD	5.62	7.14	6.86	3.66	10.82	7.15	10.60	2.10	4.15

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