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EFFECT OF CEMENT SELECTION ON RETENTION OF ZIRCONIA AND
LITHIUM DISILICATE CROWNS

by

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A THESIS

Submitted to the graduate faculty of The University of Alabama at Birmingham,
in partial fulfillment of the requirements for the degree of MS in Dentistry

BIRMINGHAM, ALABAMA

2020

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EFFECT OF CEMENT SELECTION ON RETENTION OF ZIRCONIA AND LITHIUM DISILICATE CROWNS

CHAN-TE HUANG

MASTER OF SCIENCE IN DENTISTRY

ABSTRACT

Background: Ceramic crowns (lithium disilicate and zirconia) have become the most prescribed material for the restoration of anterior and posterior teeth due to the esthetic concerns of patients and the high cost of noble alloys. Different types of cements are used for luting crowns such as zinc phosphate, glass ionomer, resin modified glass ionomer (RMGI), resin cements and bioactive cements. Selection of different cements and bonding protocols for zirconia or lithium disilicate crowns to increase their bonding strength remains a clinical challenge for dentists.

Objectives: To measure the retention strength of lithium disilicate and zirconia crowns cemented with different cements.

Methods: Extracted human premolars were mounted in acrylic and prepared to uniform dimensions with a flat-end tapered diamond bur in a lathe. The surface area of the prepared surface was calculated with digital microscopy. The teeth were scanned with a True Definition Scanner (3M). Lithium disilicate crowns or zirconia crowns were milled and crystallized or sintered following manufacturers recommendations. For lithium disilicate crowns, the intaglio surfaces were etched with 5% hydrofluoric acid for 20 seconds. All zirconia crowns were airborne particle abraded with 50 microns alumina at 2 bar pressure for 10 seconds. The adhesive and cement were applied to the teeth and crowns

according to manufacturer's instructions. Crowns were allowed to self-cure under a 2.5 kg weight, stored in a moist bag for 24 hours at 37°C and then thermocycled for 10,000 cycles from 5-50°C with a 30 second dwell time. The specimens were placed in a custom fixture in a universal testing machine and loaded in tension at a crosshead speed of 5mm/min until debonding. The tensile strength (MPa) at debonding was calculated using the maximum recorded tensile force and surface area of the preparation. Data were compared with a 1-way ANOVA and Tukey analysis ($\alpha=0.05$).

Results: Part 1 - The self-adhesive resin cement demonstrated greater retention compared to the bioactive, GI and RMGI cements. The bioactive cements had similar retention strength as RMGI cements.

Part 2 - Significant differences between the experimental and five different resin cements were noted with 1-way ANOVA ($p<.01$). The Tukey post-analysis determined that the experimental resin cement showed no difference in retention strength compared to Link Force, Calibra Ceram, or Variolink Esthetic but higher retention strength than Nexus 3 and Panavia V5. Panavia V5 was also significantly less retentive than Nexus 3. A large number of tooth fractures (60%) were noted during crown retention testing when stronger resin cements were used.

Part 3 - For cementing zirconia crowns, the RMGI cement had no significant difference in retention strength compared to the self-adhesive resin cements or the resin cement used with an adhesive. For cementing lithium disilicate crowns, the RMGI cement had significantly lower retention strength compared to the self-adhesive resin cements and the resin cement used with an adhesive. A novel self-adhesive resin cement (Panavia SA Universal) used on lithium disilicate crowns without silane application achieved

statistically similar retention strength as another self-adhesive resin cement with silane application, albeit the retention strength was numerically lower.

Conclusions: Within the limitations of the present study, different cements could provide different retention strength. In general, resin cement provided higher retention than RMGI cement when used with lithium disilicate crowns. With the use of zirconia crowns, the difference in retention between RMGI and resin cements was less pronounced. Clinicians should choose different cements based on crown retention requirements, type of ceramics and also consider other factors such as shade, translucency, strength of crowns, isolation, microleakages, resistant forms and so on.

Keywords: Crown retention strength, bioactive, zirconia, lithium disilicate, dental cements.

DEDICATION

This thesis is dedicated to my parents. Their constant encouragement, giving me strength, constitute who I am today. Without them, I would not have achieved anything I have now. To my lovely wife, who supports everything behind me, letting me keep moving forward without hesitation. To my kids, their smiles always melt my heart and become the impetus that fulfills my life.

ACKNOWLEDGMENTS

This project would not have been possible without the support of many people. First and foremost, I would like to thank my mentor, Dr. Nathaniel Lawson, who guided me through all these days, building up the ideas within this thesis, helping deal with the statistics in this thesis, solving many problems encountered during my residency, giving me opportunities to get involved in or even get awards through national or international meetings, and most importantly always thinking about how to help his residents, including me. I am fortunate to have Dr. Lawson to be my mentor through this two-years biomaterials program, without him, I could not do my best to accomplish this thesis.

I would like to thank Dr. John Burgess, who is full of knowledge, always willing to teach us and help us, instill into us enormous knowledge about biomaterials, and instruct me with the skills to formulate a qualified article.

I would like to thank Dr. Perng-Ru Liu, who helped me a lot through the course of my program, gave me the opportunities to get involved in learning clinical and teaching skills here, and also helped me with the crown milling process in this thesis.

I would like to thank Dr. Jack Lemons, who always taught and broadened us in the knowledge of biomaterials by his insight, his patience, and his enthusiasm.

I would like to thank Dr. Amjad Javed, who directed me through the correct path, encouraged me and gave me any information I needed to finish my graduate program.

I would like to thank our former and current residents, Faimeena Farheen, Anvita Maharishi, Mohammed Badahman, Yasko Darkoue, Sarah Alhalees and Shantanu Mankar, who cared for me and accompanied me through my research work.

I also would like to thank Faimeena Farheen for helping me prepare for my studies in classes and Mohammed Badahman for instructing me with some hints for thesis writing.

I would like to thank Mrs. La Tara Rogers, Sheila Rahim, and Phelecia Jemison for supporting me and accompanying me through my program and helping with collecting or ordering the materials which I needed in this thesis.

I would like to thank Stephen Liu, who is a graduate prosthodontic resident, for helping me finish parts of my thesis work including collecting, preparing, cementing, and pulling the specimens.

I would like to thank Mr. Wen Hsin Chiou and Mr. Zheng Ting Chiu from Dong Li Dental Technology CO., LTD. in California and in Taiwan for helping mill parts of the e. Max and Zirconia crowns used in this thesis.

I would like to thank Mr. James Bradley for instructing me in the use of crown designing software and helping with some of the laboratory work in this thesis.

I would like to thank Dentsply Sirona, 3M ESPE, GC America, Kuraray Corp. for sponsoring parts of this thesis and offering guidance to use their materials.

Last, I would like to thank the Chang-Gung Memorial Hospital in Taiwan to offer me this precious opportunity to further study here in the United States to get my Master's degree.

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LIST OF ABBREVIATIONS

10-MDP – 10-methacryloxydecyl dihydrogen phosphate

GI – Glass ionomer

HF acid – Hydrofluoric acid

IFU – Instructions for use

RMGI – Resin-modified glass ionomer

ZPC – Zinc phosphate cement

1. INTRODUCTION

Ceramic materials have become the preferred prosthetic restoration in many clinical situations because of adequate mechanical properties, good biocompatibility, and excellent esthetics.¹ Currently, the two ceramic materials gaining most attention from dentists are zirconia and lithium disilicate.² According to a National Dental Practice-Based Research Network survey, dentist chose lithium disilicate most frequently for anterior single-unit crowns and full zirconia for posterior single-unit crowns.³ Although PFM crowns were chosen comparably as full zirconia for posterior restorations in the survey, the trend towards using full zirconia crown is evident. Additionally, a systematic review comparing zirconia and lithium disilicate all ceramic restorations to metal-ceramic tooth restorations reported similar 5-year survival rates for single anterior and posterior crowns.⁴

Despite the high strength of the first generation of zirconia (3 mol% yttria containing with 0.25wt% alumina), also known as frame zirconia, its opacity led to the modification of dental zirconia to improve its translucency. First, by changing its sintering temperature, conventional zirconia could be rendered more translucent.⁵ By reducing the number and grain size of alumina oxide grains and relocating them within the grain structure, the second generation zirconia (3 mol% yttria, containing 0.05wt% alumina) achieved improved translucency, making it look more natural.⁶ Furthermore, as the continuous demands for esthetic restorations increased, the third generation of zirconia was developed in 2015, modified from the original 3 mol% yttria containing through increasing

the yttrium oxide contents in zirconia to 4 mol% (4Y) or 5 mol% (5Y) yttria-containing zirconia.⁷ By increasing yttria content, the third generation zirconia is composed of up to 53% cubic-phase zirconia. The third generation zirconia has the highest translucency among all zirconia but as a result, lost its transformation toughening characteristics.⁸ Therefore, the third generation of zirconia achieved higher esthetic and optical properties; at the expense of its mechanical properties.

Due to concerns of veneering porcelain chipping on zirconia frameworks,⁹ monolithic zirconia is more frequently prescribed than layered zirconia. Despite improvements in translucency, even 3rd generation zirconia lack the esthetics of glass-ceramics owing to its oxide-forming crystalline structure and high light refraction index.⁸ Therefore, zirconia is not frequently utilized in anterior aspects of oral dentition.⁷

On the contrary, glass-ceramic products made from lithium disilicate such as e.max (Ivoclar Vivadent) have very good translucency and optical performance, making them popular for anterior esthetic restorations such as veneers. The mechanical properties of lithium disilicate, although not as high as those of zirconia, are sufficient to allow monolithic posterior restorations if the thickness of the restoration proper.

Another concern of zirconia is its possible transformation from tetragonal phase to monoclinic phase in a specific environment. The aging effect, also called low-temperature degradation, could adversely reduce the mechanical strength of zirconia. The stabilizer type and content, the residual stress, and the grain size are main factors influencing this aging phenomenon.¹⁰ A slow tetragonal to monoclinic transformation occurs when 3 mol% yttria-containing zirconia is in contact with water or vapor, body fluid or during steam sterilization, which possibly leads to surface damage.¹¹ On the other hand, with the 4 or 5

mol% yttria-containing high-translucent zirconia, the cubic phase of zirconia will exist in a greater ratio which presumably results in less low-temperature degradation.

Bonding zirconia to resin cement is a clinical concern. The intrinsic properties of zirconia are not like glass-based restorations in which crystals are formed within a glass matrix. Zirconia, an oxide ceramic, cannot be effectively acid-etched to obtain a rough surface like glass ceramics. Additionally, the chemical bonding technique using conventional silane does not bond to a zirconia surface due to the absence of silica in the zirconia microstructure.¹²

Different approaches to improve zirconia bonding to cements include surface abrasion or roughening, application of a tribochemical silica coating, silica coating techniques, chloro-silane treatment, selective infiltration etching, nanostructured alumina coating, and application of phosphate ester primers or phosphate modified resin cements. Other surface treatments have also been attempted including hot chemical etching solution, laser application, zirconia ceramic powder coating, and gas-phase fluorination process are also try to positively modify the zirconia surface characteristics.¹³ By and large, many of these measures are still questionable and need further long-term evaluation.

In the early 2000's, an efficient method to chemically bond to zirconia was developed by Kuraray Co. by using phosphate monomers.¹³ This composition, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) could effectively bond to resin surface in many studies. The bonding of the phosphate group in 10-MDP formed a chemically-stable covalent bond to a zirconia surface ($-P-O-Zr$), the same as that of an organophosphate, octylphosphate.¹⁴ By 2012, the patent for 10-MDP expired and many companies strived to make a primer and/or resin cement containing 10-MDP.

A systematic review and meta-analysis of laboratory studies evaluating bonding effectiveness of zirconia was performed by Inokoshi et al. in 2014.¹⁵ A literature search from 1990 to 2013 was included. Regarding luting technique, it stated that the combination of mechanical and chemical pre-treatment appeared particularly crucial to obtain durable bonding to zirconia ceramics.

Tzanakakis et al. reviewed the literature from 1998 to 2014 to classify and analyze the existing methods and materials proposed to improve adhesion to zirconia surfaces.¹⁶ Although the results were difficult to compare due to inconsistencies in different study protocols and designs, some conclusions could be drawn. First, airborne-particle abrasion was a commonly used reference method included in most research protocols. Tribochemical coating (silica coated alumina) could enhance bonding if followed by a silane to increase the wetting capacity of the inorganic surface (allowing a better flow of a resin cement across the surface) and help to form a chemical bond with resin cements. Following mechanical preparation of a surface, phosphate-based adhesive monomers were necessary for chemical bonding. Surface contamination and aging had negative effects on adhesion to zirconia. Finally, the role of aging was important to most research protocol, however the relevance of aging should be confirmed by clinical trials.

In a systemic review and meta-analysis conducted by Thammajaruka et. al in 2018, the bonding of composite cements to zirconia was analyzed by comparing in-vitro studies prior 2016.¹⁷ The data analyzed included bond strength data to identify the influence that composite cements, type of test methodology, and chemical and mechanical pre-treatments had on the bond strength of composite cements to zirconia in three different artificial aging conditions. This meta-analysis indicated that in a non-aged condition, alumina air abrasion

combined with MDP-containing primer groups tended to yield the highest actual mean bond strength. In an intermediate-aged condition, tribochemical silica coating or alumina air abrasion combined with MDP-containing primer groups tended to yield the highest actual mean bond strength. In an aged condition, ceramic coating combined with MDP-containing primer groups tended to yield the highest actual mean bond strength.

In general, there is no universally accepted protocol exists for long-lasting and biologically safe zirconia bonding. However, from most current studies comparing zirconia bonding system, it can be concluded that alumina air abrasion combined with application of an MDP containing primer to zirconia surface is the simplest and most effective way to bond zirconia to composite resin.

Lithium disilicate is a kind of glass ceramic composed of a silica-rich phase. Application of hydrofluoric acid and silane is the method to enhance bonding of glass ceramic to resin composite.¹⁸ Hydrofluoric acid etching can create surface texture allowing micromechanical interlocking of composite resin cement into the lithium disilicate surface. On the other hand, silane behaves as a bridge between composite resin cement and glass ceramic by connecting both surfaces chemically via two functional groups.¹⁹ Although bonding will increase the strength of glass ceramic, it is unnecessary to bond glass ceramics in every case. When glass ceramic restorations have a thickness greater than 1.5mm, cementation with glass ionomer or resin-modified glass ionomer cement is sufficient.²⁰

Dental cements used for luting crowns permanently include zinc polycarboxylate, zinc phosphate, glass ionomer, resin modified glass ionomer (RMGI) and resin cements.²¹ These cements are mostly used to retain crowns on natural teeth by creating a strong interface between dentin and the internal surface of the restoration. The longevity of the

bond stability between the zirconia crown and luting cement and the cement and dentin is influenced by factors such as temperature, exposure to saliva, and mechanical stress during mastication. Although there is not accepted standard for measuring the retention provided by dental cements, a systematic review by Heintze analyzing 18 studies recommended reasonable conditions for crown retention tests.²²

Zinc phosphate cement can only provide mechanical retention for the tooth.²³ Glass ionomer cements set chemically through a reaction of a silicate glass powder and polyalkenoic acid. Resin modified glass ionomers are similar to glass ionomers aside from their ability to dual cure imparted through the addition of methacrylate groups and photoinitiators. The addition of the resin increases the flexural and tensile strength of the RMGI and lowers the modulus of elasticity. Through polyacrylic acid of glass ionomer or RMGI cements, ionic bonds can be formed with calcium ions in the hydroxyapatite of enamel or dentin surface.²⁴ All glass ionomer based materials contain fluoride releasing components which help in high caries risk patients.²⁵ RMGI cements generally have good clinical retention with a study reporting less than a 3% annual failure rate for a follow-up periods over 13 years.²⁶ Resin cements have stronger mechanical properties and also have lower solubility in the oral environment, reduced microleakage at the restoration-tooth interface, good optical properties and low incidence of marginal staining and recurrent caries.²⁷ Self-adhesive resin cements have also been gaining popularity and can also be used for cementation of restorations. Self-adhesives cements typically transition from an acidic pH to a neutral pH during setting. It is the initial acidic pH that is responsible for the self-etching properties. The dual-cure resin cements based on tertiary amine chemistry can

result in these materials not being color stable over years, so caution is advised when considering them under thin or translucent all ceramic restorations in the anterior.

In recent years, novel bioactive cements were developed and most companies claimed that these cements could have the ability to remineralize initial caries and prevent further secondary caries formation. Calibra Bio cement (formerly Ceramir Crown & Bridge) manufactured by Dentsply is one of the novel bioactive cement and has shown to deposit hydroxyapatite on its surface after immersion in human saliva.²⁸ Other examples of bioactive cements are Theracem by Bisco and Activa Bioactive Cement by Pulpdent.

Decision making for choosing cements can be complicated. Based on a survey from findings of the National Dental Practice-Based Research Network, dentists chose to use RMGI cements most frequently (52.2%) followed by resin cements (29.2%) for single-unit crowns.²⁹ Irrespective of any clinical factors, some dentists chose to always bond crowns with resin cement (20.3%) while other chose to never bond (39.1%). The decision to choose a resin cement instead of a glass-ionomer or RMGI cement could depend on the ability to isolate the tooth, the need for crown retention, the esthetic requirements of the case, and the strength of the crown restorative material. In situations when crown preparations are too short, tapered or otherwise lack of resistance form, bonding with an adhesives and resin cement is usually necessary to compensate for the deficiency in retention. On the other hand, if retention is sufficient, cementation with RMGI can be performed to simplify the procedure.

2. OBJECTIVES

The main objective of this dissertation is to measure the retention strength of lithium disilicate and zirconia crowns cemented with different kinds of cements. In order to address the objective of the thesis successfully, the thesis is composed of following sections.

2.1 Retention Strength of Glass Ceramic Crowns Bonded with GI, RMGI, Resin and Bioactive Cements

The objective is to measure and compare the retention strength of lithium disilicate crowns (e.max CAD) to prepared human teeth with glass ionomer, RMGI, resin, and novel bioactive cements.

2.2 Retention Strength of Glass Ceramic Crowns Bonded with Different Resin Cements

The objective is to measure and compare the retention strength of lithium silicate crowns (e.max CAD) to prepared human teeth with a novel resin cement and compare this to other clinically successful resin cements.

2.3 Retention Strength of Zirconia and Lithium Disilicate Crowns Bonded with RMGI Cement, a Self-adhesive Resin Cement, a Resin Cement with Adhesive and a Novel Self-adhesive Resin Cement

The objective is to measure retention of zirconia and lithium disilicate crowns bonded with RMGI cement (Rely X Luting Plus), a self-adhesive resin cement (Rely X Unicem 2), a resin cement with adhesive (Rely X Ultimate) and a novel self-adhesive resin cement (Panavia SA Universal).

3. NULL HYPOTHESES



1. There is no difference in the retention strength of lithium disilicate crowns to prepared human teeth cemented with glass ionomer, RMGI, resin, and bioactive cements.
2. There is no difference for the retention strength of lithium silicate crowns to prepared human teeth cemented with a novel resin cement compared to other clinically successful resin cements.
3. There is no difference for the retention strength of zirconia and lithium disilicate crowns bonded with a RMGI cement, a self-adhesive resin cement, a resin cement used with a bonding agent and a novel self-adhesive resin cement.




4. MATERIALS

4.1 Retention Strength of Glass Ceramic Crowns Bonded with GI, RMGI, Resin and Bioactive Cements

The cements used in the present study are summarized in Table 1, the glass ceramic used in the present study is IPS e.max CAD LT shade A2 (Ivoclar Vivadent)

Table 1: Trade names and pictorial representation of cements used in the study

Group	Material	Manufacturer	Type	Pictorial Representation
Group 1	Ketac Cem	3M ESPE	Glass ionomer	
Group 2	RelyX Luting Plus	3M ESPE	RMGI	

Group 3	FujiCEM 2	GC America	RMGI	
Group 4	Calibra Bio	Dentsply Sirona	Calcium aluminat e “Bioactiv e”	
Group 5	Calibra Universal	Dentsply Sirona	Self- adhesive resin	

4.2 Retention Strength of Glass Ceramic Crowns Bonded with Different Resin

Cements

The cements and primers used in the present study are summarized in Table 2, the glass ceramic used in the present study is IPS e.max CAD LT shade A2 (Ivoclar Vivadent)

Table 2: Trade names of cements and primers used in the study

Group	Manufact urer	Resin Cement	Ceramic Primer	Tooth Primer
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Group 1	Kuraray	Panavia V5	Clearfil Ceramic Primer	V5 tooth primer
Group 2	Ivoclar Vivadent	Variolink Esthetic	Monobond Plus	AdheSE Universal
Group 3	GC	Link Force	G-Multi Primer	G-Premio Bond
Group 4	Dentsply Sirona	Calibra Ceram	Calibra Silane Coupling Agent	Prime&Bond Elect
Group 5	Kerr	Nexus 3	Kerr Silane	Optibond XTR
Group 6	3M	Experimental 3M cement	Experimental 3M adhesive	Experimental 3M adhesive

4.3 Retention Strength of Zirconia and Lithium Disilicate Crowns Bonded with RMGI Cement, a Self-adhesive Resin Cement, a Resin Cement with Adhesive and a Novel Self-adhesive Resin Cement

The ceramics and cements used in the present study are summarized in Table 3.

Table 3: Trade names of ceramics and cements used in the study

Group	Manufacturer	Ceramics	Manufacturer	Cements
Group 1	Kuraray	Katana HT	3M	RelyX Luting Plus
Group 2	Kuraray	Katana HT	3M	Rely X Unicem 2
Group 3	Kuraray	Katana HT	Kuraray	Panavia SA Universal
Group 4	Kuraray	Katana HT	3M	Rely X Ultimate (with Scotch Bond Universal)
Group 5	Ivoclar Vivadent	e.max CAD LT	3M	RelyX Luting Plus
Group 6	Ivoclar Vivadent	e.max CAD LT	3M	Rely X Unicem 2
Group 7	Ivoclar Vivadent	e.max CAD LT	Kuraray	Panavia SA Universal
Group 8	Ivoclar Vivadent	e.max CAD LT	3M	Rely X Ultimate (with Scotch Bond Universal)

5. METHODS

5.1 Specimen Selection

Mandibular second premolar teeth, freshly extracted for orthodontic reasons, were collected from the Oral and Maxillofacial Surgery Department of the UAB School of Dentistry. Each group comprised of 8 teeth, so a total of 40 teeth for part 1, 64 teeth for part 2 and 64 teeth for part 3 were collected. All of these teeth were examined to be without caries, non-carious cervical lesions and cracks. Teeth were examined for cracks under 2.5X loupes.

5.2 Specimen Mounting

The roots of the selected teeth were notched with a rotating disc on a slow-speed handpiece (to aid with mounting) and the occlusal surfaces were ground flat on a model trimmer. For mounting, the teeth were centered in Teflon cylinders with the help of a surveyor and digital caliper and then embedded in clear auto-polymerizing acrylic resin (Yates Motloid, USA). (Figure 1)

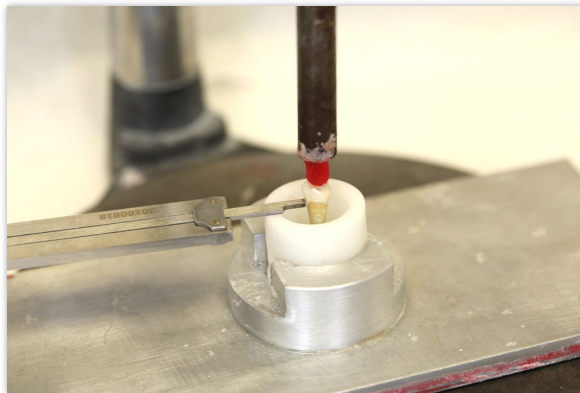


Figure 1. Tooth mounting

5.3 Standardized Tooth Preparation

After complete curing of the acrylic resin, the samples were fixed into a lathe (Central Machinery, USA.) (Figure 2) for precise uniform reduction with a diamond cutting tool to produce a uniform tooth preparation with exact taper, diameter and fit (Figure 3).

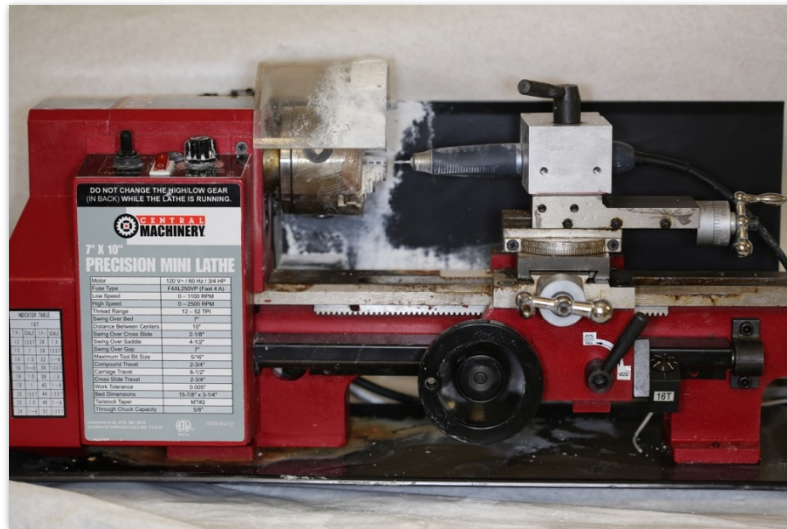


Figure 2. Lathe for preparation

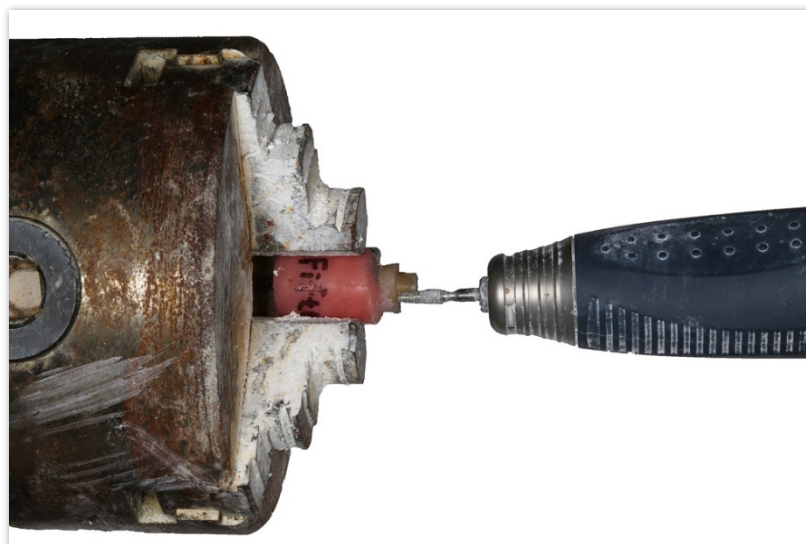


Figure 3. Standardized tooth preparation

5.3.1 Original Tooth Preparation Design

For the first and second parts of our studies, the original tooth preparation design was utilized. The teeth were prepared to uniform dimensions (16° total taper using 846.11.025 HP medium flat end taper diamond bur, Brasseler, USA, and 3mm preparation height using X889 diamond bur). (Figure 4) Teeth were kept moist prior to bonding.

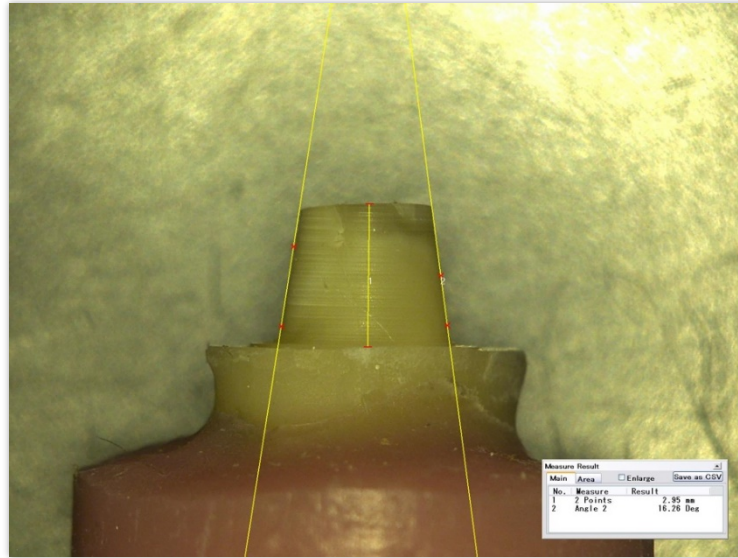


Figure 4. Original tooth preparation design

5.3.2 Modified Tooth Preparation Design

For the third part of the study, a modified tooth preparation design was utilized. The teeth were prepared to uniform dimensions (23° total taper using 846.11.025 HP medium flat end taper diamond bur, Brasseler, USA, and 2mm preparation height using X889 diamond bur). (Figure 5) In order to standardize the area of the margin, a secondary preparation was performed to standardize the width of the margin to 1mm. (Figure 6) Teeth were kept moist prior to bonding.

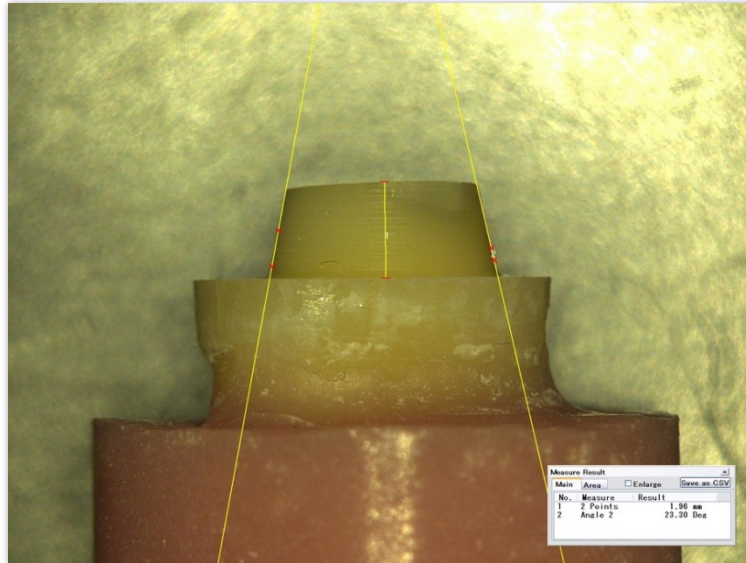


Figure 5. Modified tooth preparation design

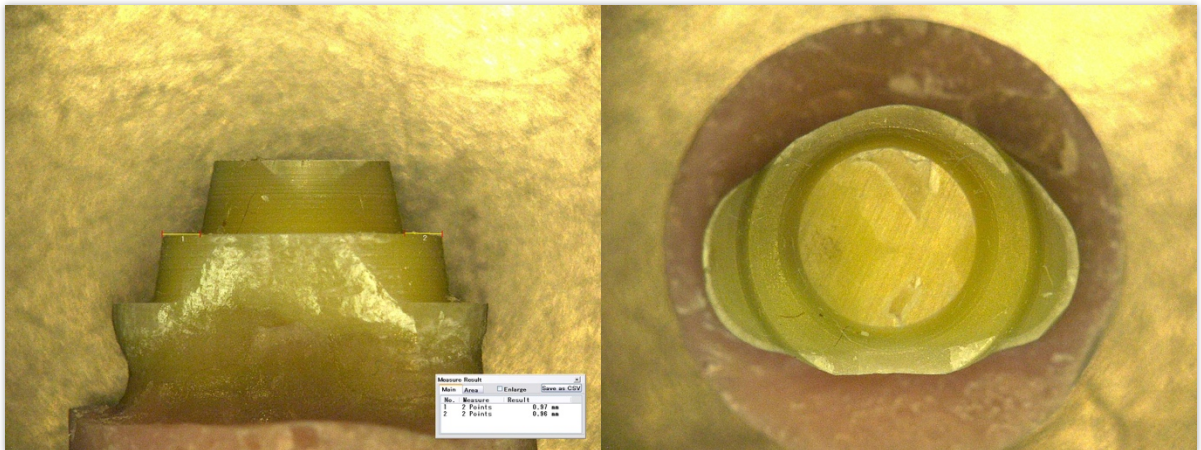


Figure 6. Tooth after secondary preparation

5.4 Total Bonding Surface Area Calculation

The bonding surface area of the prepared surface was calculated under 20X in a digital microscope (Keyence; Keyence Co, Japan) (Figure 7 and Figure 8) In order to

calculate the bonding surface area, the lateral walls, occlusal table, and margin width were all measured separately. The bonding area was calculated using the formula:

Total bonding area = lateral surface area of truncate cone + area of top circle of truncated cone + (Difference between area of base and bottom circle of truncated cone)

(Figure 9)

Each of these areas will be calculated as described below:

1. Lateral surface area of the truncated cone is calculated by formula = $\pi (R + r) S$ sq. units
2. The area of top circle of truncated cone was measured as πr^2 . This area represents the occlusal table of the preparation.
3. The base of the preparation was measured with a function in the digital microscope (Keyence; Keyence Co, Japan), The bottom circle of the truncated cone was measured as πR^2 . The difference between these values represents the width of the preparation margin.



Figure 7. Keyence digital microscope

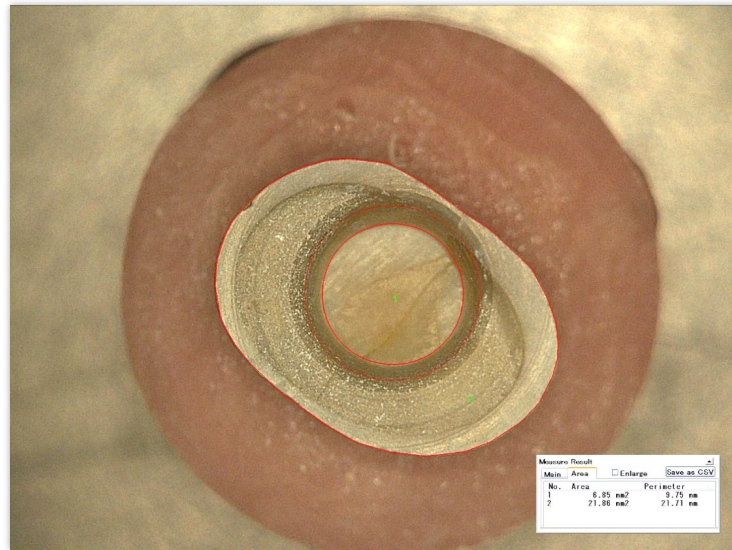


Figure 8. Area measurement

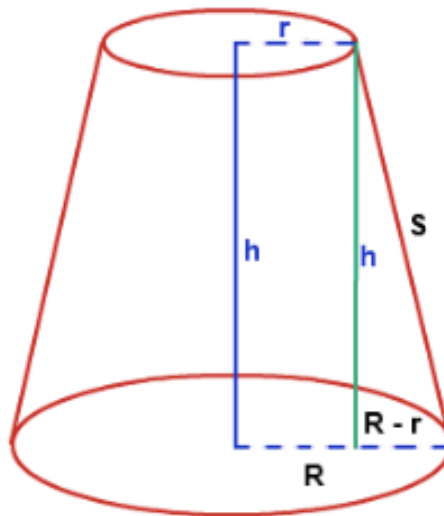


Figure 9. Surface area calculation

5.5 Preparation of Crowns

All tooth preparations were scanned with a digital intraoral scanner (3M Lava True Definition Scanner; 3M Co., MN, USA). The .stl files were sent to the laboratory to have

the crowns milled from lithium disilicate or zirconia. The crowns were designed with cement spaces of 10 μm for zirconia crowns and 40 μm for lithium disilicate crowns based on manufacturer's recommendation by 3shape Dental System (3shape Co., Copenhagen, Denmark). The crowns were designed with a flat bar which allowed the crowns to be pulled off with a wire loop. (Figure 10) Lithium disilicate crowns were crystallized and zirconia crowns were sintered according to the manufacturer's recommendations.

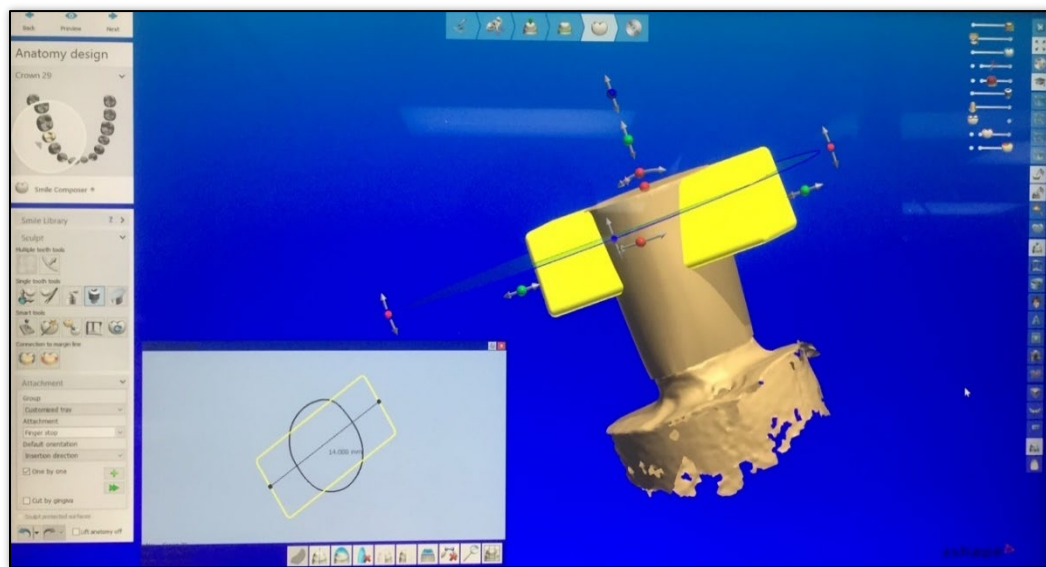


Figure 10. Crown design

5.6 Cementation of Crowns

5.6.1 Retention Strength of Glass Ceramic Crowns Bonded with GI, RMGI, Resin Bioactive Cements

All tooth preparations were thoroughly cleaned with water and blotted dry prior to cementation. All lithium disilicate crowns were etched with 5% hydrofluoric acid (Vita) for 20 seconds. The adhesive and cement were applied to the teeth and crowns according

to manufacturer's instructions. The crowns cemented with Calibra Universal had 1 coat of Calibra silane applied prior to bonding. The crowns were then seated with finger pressure to ensure a complete seating. (Figure 11) Excess cement was carefully removed with a microbrush or sponge in an uncured stage while the crown was held fixed. They were immediately placed under a 2.5kg load. All crowns were self-cured only for the amount of time reported in their instructions for use (IFU).

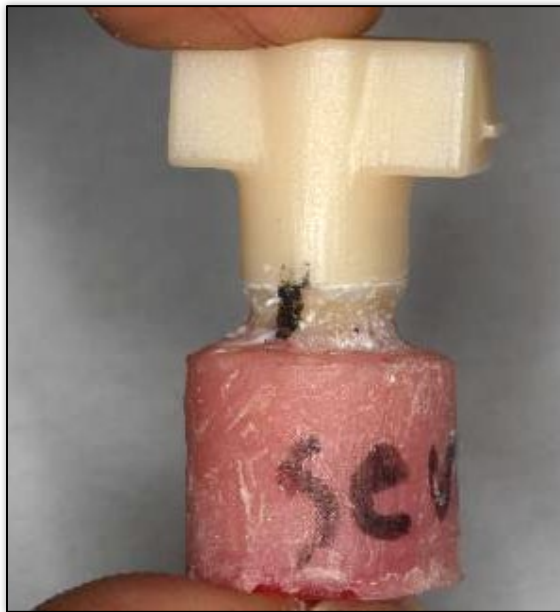


Figure 11. Cementation of crowns

5.6.2 Retention Strength of Glass Ceramic Crowns Bonded with Different Resin Cements

All tooth preparations were thoroughly cleaned with water and blotted dry prior to cementation. All tooth surfaces were treated in the self-etching mode and bonded with adhesives according to the manufacturer's IFU:

- For Kuraray group, the tooth surfaces received Clearfil Tooth Primer for 20 seconds then dried.
- For Ivoclar Vivadent group, the tooth surfaces received AdheSE Universal for 20 seconds, dried then light cured for 10 secs.
- For GC group, the tooth surfaces received G-Premio Bond for 10 seconds, dried for 5 secs then light cured for 10 secs.
- For Dentsply Sirona group, the tooth surfaces received Prime&Bond Elect for 20 secs, dried for 5 seconds then light cured for 10 secs.
- For Kerr group, the tooth surfaces were primed first with OptiBond XTR Primer for 20 secs, dried for 5 secs then bonded with OptiBond XTR Adhesive for 15 seconds and dried for 15 secs.
- For 3M group, the tooth surfaces received experimental 3M adhesive for 20 secs, dried for 5 secs then light cured for 10 secs.

The lithium disilicate crowns were etched with 5% hydrofluoric acid for 20 seconds (Ceramic etching gel, Ivoclar Vivadent). Then the ceramic primer and the resin cement was applied to the crowns according to manufacturer's IFU:

- For Kuraray group, the crowns were silanized by Clearfil Ceramic Primer, dried then cemented with Panavia V5. The cement was allowed to self-cure for 3 mins.
- For Ivoclar Vivadent group, the crowns were silanized by Monobond Plus for 60 secs, dried then cemented with Variolink Esthetic. The cement was allowed to self-cure for 5 mins.

- For GC group, the crowns were silanized by G-Multi Primer, dried then cemented with LinkForce. The cement was allowed to self-cure for 4 mins.
- For Dentsply Sirona group, the crowns were silanized by Calibra Silane Coupling Agent, dried then cemented with Calibra Ceram. The cement was allowed to self-cure for 5 mins.
- For Kerr group, the crowns were silanized by Kerr Silane, dried then cemented with NX3 Nexus. The cement was allowed to self-cure for 6 mins.
- For 3M group, the crowns were silanized by experimental 3M adhesive for 20 secs, dried for 5 secs then cemented with experimental 3M cement. The cement was allowed to self-cure for 6 mins.

The crowns were seated with finger pressure to ensure a complete seating. Excess cement was carefully removed with a microbrush or sponge in an uncured stage while the crown was being held fixed. They were immediately placed under a 2.5kg load and a glycerine gel was placed around the margins. The 3M crowns were rinsed with water after cure. All crowns were self-cured only for the amount of time reported in their IFU.

5.6.3 Retention Strength of Zirconia and Lithium Disilicate Crowns Bonded with RMGI Cement, a Self-adhesive Resin Cement, a Resin Cement with Adhesive and a Novel Self-adhesive Resin Cement

All tooth preparations were thoroughly cleaned with water and blotted dry prior to cementation. All lithium disilicate crowns were etched with 5% hydrofluoric acid (Vita) for 20 seconds and silanized (Calibra[®] silane; Dentsply Caulk, DE, USA) for 20 seconds

except for RMGI group. The group for the novel self-adhesive resin cement (Panavia SA Universal; Kuraray Co., Japan) skipped the step for applying silane. All zirconia crowns were airborne particle abraded with 50 microns alumina at 2 bar pressure for 10 seconds. The adhesive (Scotchbond Universal, 3M ESPE, Germany) and cement were applied to the teeth and crowns according to manufacturer's instructions. The crowns were then seated with finger pressure to ensure a complete seating. Excess cement was carefully removed with a microbrush or sponge in an uncured stage while crown was held fixed. They were immediately placed under a 2.5kg load. All crowns were self-cured only for the amount of time reported in their IFU.

5.7 Storage, Load Cycling and Thermocycling

5.7.1 Retention Strength of Glass Ceramic Crowns Bonded with GI, RMGI, Resin Bioactive Cements

The crowns were stored in a moist zip lock bag for 24 hours at 37°C. Afterwards they were stored in phosphate-buffered saline (PBS) at 37°C for 30 days. Then 10,000 thermocycles between 5 and 55°C with dwell time for 30 secs was performed.

5.7.2 Retention Strength of Glass Ceramic Crowns Bonded with Different Resin Cements

The crowns were stored in a moist zip lock bag for 24 hours at 37°C. The crowns were then thermocycled for 10,000 cycles between 5 and 55°C with dwell time of 30 secs. Afterwards samples were stored in water at 37°C for 30 days.

5.7.3 Retention Strength of Zirconia and Lithium Disilicate Crowns Bonded with RMGI Cement, a Self-adhesive Resin Cement, a Resin Cement with Adhesive and a Novel Self-adhesive Resin Cement

The crowns were stored in a moist zip lock bag for 24 hours at 37°C. The crowns were then thermocycled for 30,000 cycles between 5 and 55°C with dwell time of 30 secs.

5.8 Tensile Retention Testing

The specimens were fixed in a universal testing machine (INSTRON model 5565; Instron Corp, Norwood, MA, USA) and loaded in tension at a crosshead speed of 5 mm/min until debonding. (Figure 12) The crowns were grasped by two wings on the sides of the crowns. The force (N) of debonding was recorded. The retention force was calculated in MPa by the formula:

$$\text{Retention (MPa)} = \frac{\text{Debonding Force (N)}}{\text{Total Bonding Surface area of Preparation (mm}^2\text{)}}$$



Figure 12. Crown retention test

5.9 Failure Mode Analysis

Examination of the failure site was made optically with a digital microscope (Keyence; Keyence Co, Japan) at 100 X and categorized according to Table 4.

Table 4. Mode of failures

Category	Description
Category 1	Cement mainly on prepared tooth (over 75%)
Category 2	Cement on both crown and tooth (between 25 and 75%)
Category 3	Cement mainly on crown (over 75%)
Category 4	Fracture of tooth root without crown separation
Category 5	Fracture of Crown

6. STATISTICAL ANALYSIS

A one-way ANOVA was used for part 1 and part 2 studies to analyze the retention force or stress between all different groups and check if there was any difference. ($p < .001$).

A two-way ANOVA was performed to compare the retention force or stress between all different ceramics and cements for part 3 study to check if there was any difference. ($p < .001$).

Tukey post-hoc analysis was used to compare and divide all groups into statistically similar groups.

Statistical analyses were conducted using the SPSS® computer software system, release 25 (IBM SPSS Statistics; IBM Co., NY, USA).

7. RESULTS

7.1 Retention Strength of Glass Ceramic Crowns Bonded with GI, RMGI, Resin and Novel GI Hybrid Cements

7.1.1 Crown Retention Strength

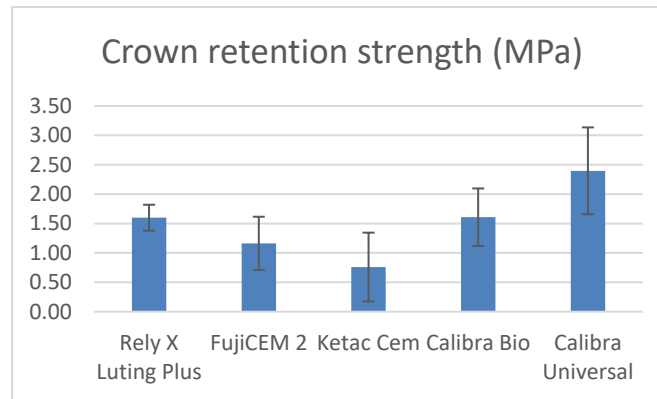


Figure 13. Crown retention strength of different kinds of cements for bonding e.Max crowns.

7.1.2 Statistical Analysis

A 1-way ANOVA found significant differences between cements.

Table 5. ANOVA result of different kinds of cements for bonding e.Max crowns.

ANOVA					
Retention Strength					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	14.038	4	3.510	13.042	.000
Within Groups	11.840	44	.269		
Total	25.879	48			

A Tukey post-hoc analysis group materials into statistically significantly different groups.

Table 6. Tukey post-hoc analysis of different kinds of cements for bonding e.Max crowns.

Retention_Strength				
Tukey HSD ^{a,b}				
Cement	N	Subset for alpha = 0.05		
		1	2	3
Ketac Cem	10	.7590		
FujiCEM 2	10	1.1610	1.1610	
Rely X Luting Plus	10		1.5980	
Calibra Bio	10		1.6060	
Calibra Universal	9			2.3944
Sig.		.436	.334	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 9.783.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

7.1.3 Failure Mode Analysis

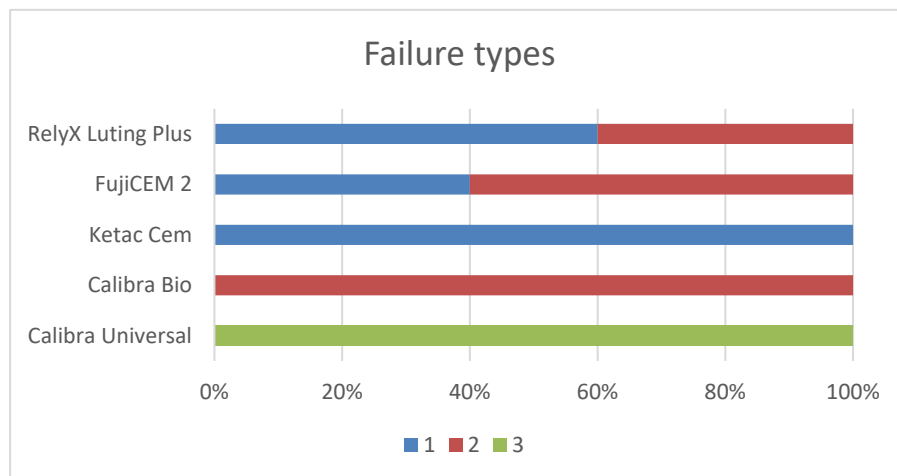


Figure 14. Failure mode analysis of different kinds of cements for bonding e.Max crowns.

7.2 Retention Strength of Glass Ceramic Crowns Bonded with Different Resin Cements

7.2.1 Crown Retention Strength

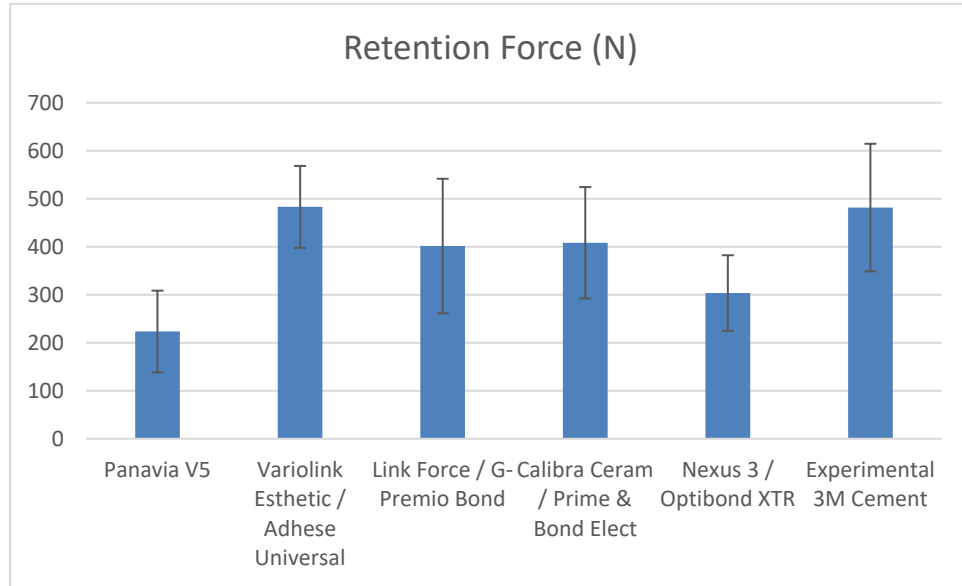


Figure 15. Crown retention force of different resin cements for bonding e.Max crowns.

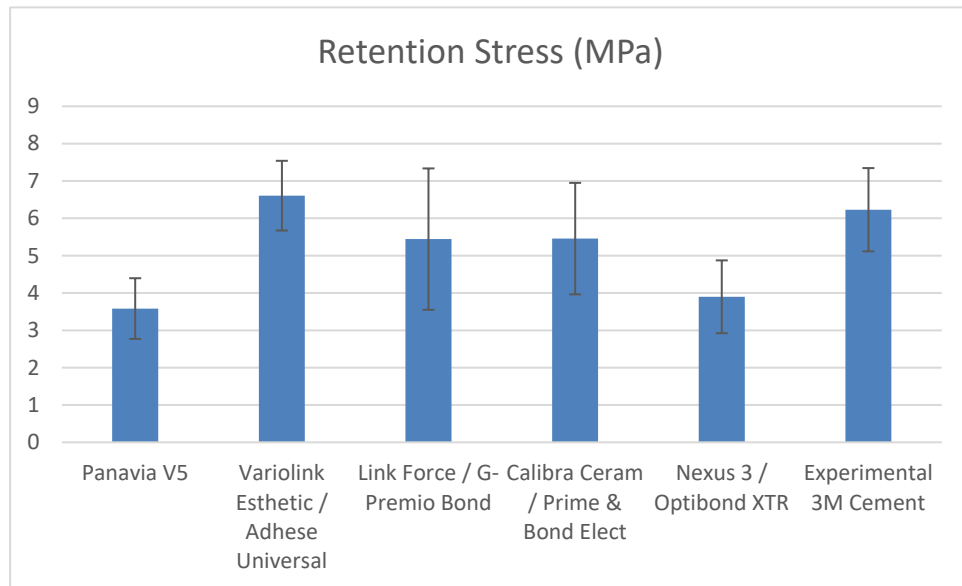


Figure 16. Crown retention stress of different resin cements for bonding e.Max crowns.

7.2.2 Statistical Analysis

A 1-way ANOVA found significant differences between cements.

Table 7. ANOVA result of retention force of different resin cements for bonding e.Max crowns.

ANOVA					
Forces	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	413596.254	5	82719.251	6.862	.000
Within Groups	494255.571	41	12055.014		
Total	907851.825	46			

A Tukey post-hoc analysis group materials into statistically significantly different groups.

Table 8. Tukey post-hoc analysis of retention force of different resin cements for bonding e.Max crowns.

Forces				
Tukey HSD ^{a,b}				
Materials	N	Subset for alpha = 0.05		
		1	2	3
Panavia V5	8	223.4687		
Nexus 3	7	303.4657	303.4657	
Link Force	8		401.4688	401.4688
Calibra Ceram	8		408.3225	408.3225
Experimental 3M	8			481.7188
Variolink Esthetic	8			483.0875
Sig.		.703	.424	.685

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 7.814.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Table 9. ANOVA result of retention stress of different resin cements for bonding e.Max crowns.

ANOVA					
Stress	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	69.105	5	13.821	7.724	.000
Within Groups	73.359	41	1.789		
Total	142.464	46			

A Tukey post-hoc analysis group materials into statistically significantly different groups.

Table 10. Tukey post-hoc analysis of retention stress of different resin cements for bonding e.Max crowns.

Stress				
Tukey HSD ^{a,b}				
Materials	N	Subset for alpha = 0.05		
		1	2	3
Panavia V5	8	3.2004		
Nexus 3	7	3.8984	3.8984	
Link Force	8		5.4430	5.4430
Calibra Ceram	8		5.4562	5.4562
Experimental 3M	8			6.2299
Variolink Esthetic	8			6.6068
Sig.		.905	.217	.527

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 7.814.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

7.2.3 Failure Mode Analysis

60 % of teeth (28 out of 48) were fractured during the retention strength test.

A typical fractured tooth after crown retention test was illustrated in Figure 18.

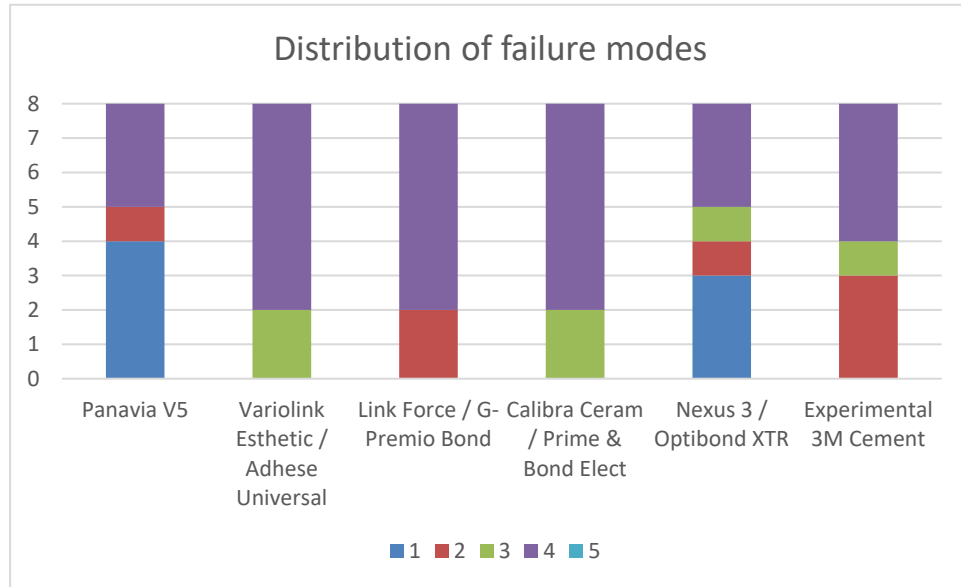


Figure 17. Failure mode analysis of different resin cements for bonding e.Max crowns.



Figure 18. A typical fractured tooth after crown retention test.

7.3 Retention Strength of Zirconia and Lithium Disilicate Crowns Bonded with RMGI Cement, a Self-adhesive Resin Cement, a Resin Cement with Adhesive and a Novel Self-adhesive Resin Cement

7.3.1 Crown Retention Stress

The crown retention stress (MPa) for all groups are presented in Table 11 and Figure 19 below.

Table 11. Crown retention stress (MPa) of different cements for bonding zirconia and e.Max crowns.

Group	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
Ceramic	Zirconia	Zirconia	Zirconia	Zirconia	e. max	e. max	e. max	e. max
Cement	3M RelyX Luting Plus	3M RelyX Unicem 2	Kuraray Panavia SA Universal	3M RelyX Ultimate	3M RelyX Luting Plus	3M RelyX Unicem 2	Kuraray Panavia SA Universal	3M RelyX Ultimate
1	3.10	7.60	3.55	3.96	0.65	5.87	3.82	6.59
2	6.09	2.36		8.71	1.44	5.32	3.01	11.59
3	2.66	2.22	5.42	7.72	0.88	8.17	3.35	8.46
4	3.73	5.82	5.35	2.72	0.96	5.39	4.43	4.69
5	1.74	5.72	4.42	3.07	1.63	9.06	7.94	9.38
6	3.75	3.44	4.34	6.47	0.66	5.79	4.35	7.31
7	2.26		6.28	4.28	0.88	6.09	5.89	5.83
8	4.38		2.17	1.79	0.33	4.46	2.62	2.39
AVG	3.463259	4.52458	4.506261	4.838302	0.931237	6.269549	4.427088	7.03099
St Dev	1.368439	2.177782	1.359848	2.505608	0.425649	1.54757	1.744953	2.85855 3

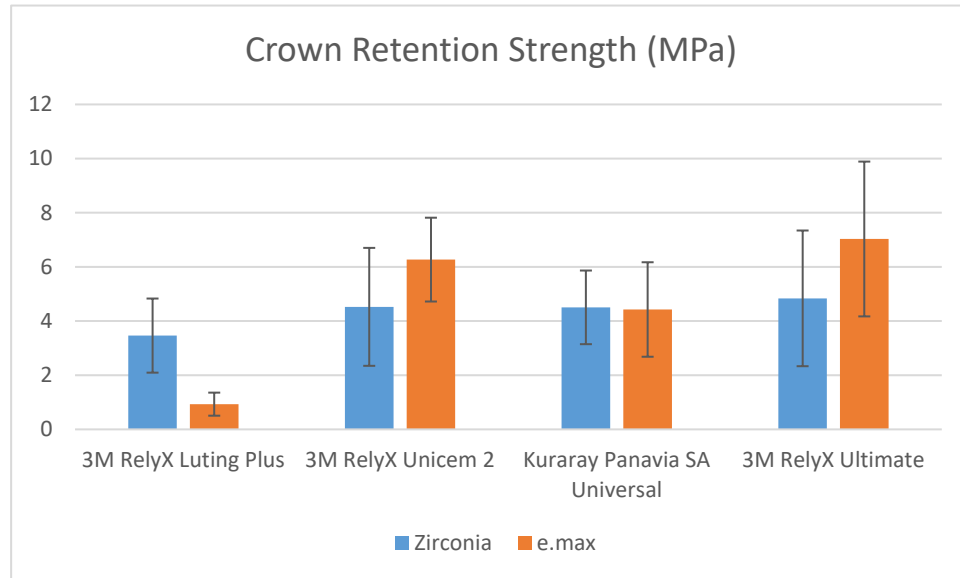


Figure 19. Crown retention stress (MPa) of different cements for bonding zirconia and e.Max crowns.

A 2 way ANOVA was performed to compare the crown retention stress for factors cement type (RelyX Luting Plus, RelyX Unicem, Panavia SA Universal and Rely X Ultimate) and ceramic type (zirconia and lithium disilicate). The factor “ceramic” was not significant ($p=0.50$), however the factor “cement” and the interaction between “ceramic” and “cement” were both significant.

Table 12. 2 way ANOVA of the crown retention stress for factors cement type and ceramic type.

Tests of Between-Subjects Effects (2-way ANOVA)

Dependent Variable: CTstress

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	187.828 ^a	7	26.833	7.553	.000
Intercept	1222.406	1	1222.406	344.107	.000
Ceramic	1.642	1	1.642	.462	.500

CTcement	128.861	3	42.954	12.091	.000
Ceramic * CTcement	54.097	3	18.032	5.076	.004
Error	188.277	53	3.552		
Total	1610.006	61			
Corrected Total	376.106	60			

a. R Squared = .499 (Adjusted R Squared = .433)

Due to the significant interaction, separate 1-way ANOVAs were performed to compare cements for both zirconia and lithium disilicate. The 1-way ANOVA for difference cements with zirconia crowns was not significant ($p=.52$).

Table 13. 1 way ANOVA for difference cements with zirconia crowns.

1-way ANOVA

Strength (zirconia)

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	8.522	3	2.841	.773	.520
Within Groups	91.873	25	3.675		
Total	100.395	28			

The 1-way ANOVA for difference cements with lithium disilicate crowns was significant ($p<.01$).

Table 14. 1 way ANOVA for difference cements with lithium disilicate crowns.

1-way ANOVA

Strength (lithium disilicate)

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	177.454	3	59.151	17.180	.000
Within Groups	96.404	28	3.443		
Total	273.859	31			

For lithium disilicate, the Tukey post-hoc analysis determined that Luting Plus was significantly less retentive than all other materials. The Rely X Ultimate was statistically similar to Rely X Unicem but more retentive than Panavia SA Universal.

Table 15. Tukey post-hoc analysis for difference cements with lithium disilicate crowns.

Tukey post-hoc analysis

Strength (lithium disilicate)

CTcement	N	Subset for alpha = 0.05		
		1	2	3
Luting Plus	8	.9288		
Panavia	8		4.4263	
Unicem	8		6.2688	6.2688
Ultimate	8			7.0300
Sig.		1.000	.217	.844

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 8.000.

7.3.2 Crown Retention Force

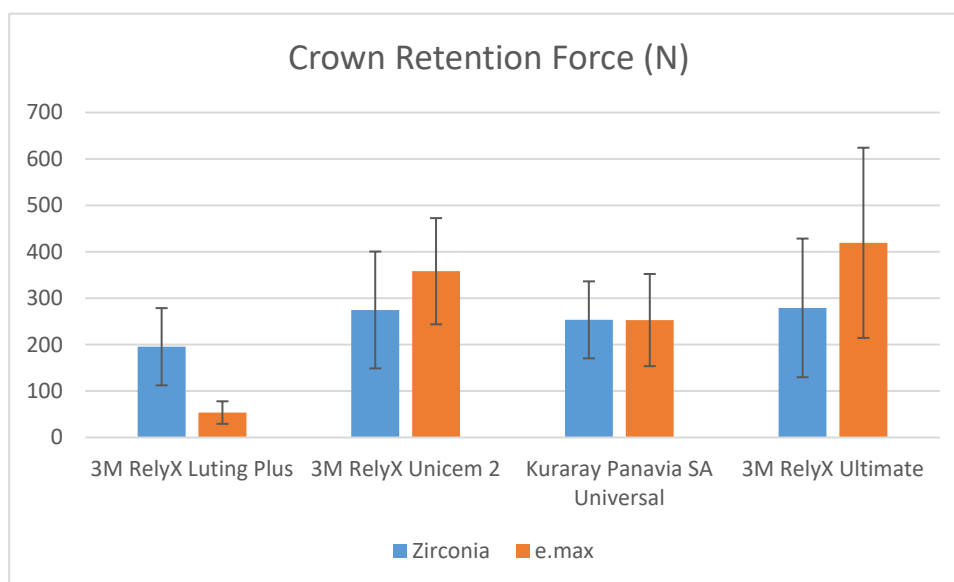
The crown retention force for all groups are presented in Table 16 and Figure 20 below.

Table 16. Crown retention force (N) of different cements for bonding zirconia and e.Max crowns.

Group	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
Ceramic	Zirconia	Zirconia	Zirconia	Zirconia	e. max	e. max	e. max	e. max
Cement	3M RelyX Luting Plus	3M RelyX Unicem 2	Kuraray Panavia SA Universal	3M RelyX Ultimate	3M RelyX Luting Plus	3M RelyX Unicem 2	Kuraray Panavia SA Universal	3M RelyX Ultimate
1	353.82	425.98		379.34	15.09	387.39	170.84	705.6
2	130.24	136.98	268.08	232.07	33.02	173.63	282.74	543.69

3	137.26		263.88	244.17	79.81	271.32	362.26	358.45
4	173.21	152.79	300.43	135.21	90.95	310.09	284.34	308.46
5	270.06	413.08	367.44	434.91	54.15	395.87	262.15	81.37
6	157.06		180.42	74.13	43.1	567.29	99.02	303.11
7	111.97	221.43	280.46	224.89	58.55	399.13	173.7	405.5
8	230.51	298.01	113.26	508.6	54.82	360.83	388.45	648.58
AVG	195.52	274.71	253.42	279.17	53.69	358.19	252.94	419.35
St Dev	83.20	125.89	82.91	149.32	24.28	114.39	99.27	204.90

Figure 20. Crown retention force (N) of different cements for bonding zirconia and e.Max crowns.



A 2 way ANOVA was performed to compare the crown retention force for factors cement type (RelyX Luting Plus, RelyX Unicem, Panavia SA Universal and Rely X Ultimate) and ceramic type (zirconia and lithium disilicate). The factor “ceramic” was not significant ($p=0.50$), however the factor “cement” and the interaction between “ceramic” and “cement” were both significant.

Table 17. 2 way ANOVA of the crown retention force for factors cement type and ceramic type.

Tests of Between-Subjects Effects (2-way ANOVA)

Dependent Variable: CTforce

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	658971.718 ^a	7	94138.817	6.367	.000
Intercept	4110794.150	1	4110794.150	278.035	.000
Ceramic	6245.313	1	6245.313	.422	.519
CTcement	465117.340	3	155039.113	10.486	.000
Ceramic * CTcement	177870.038	3	59290.013	4.010	.012
Error	783614.150	53	14785.173		
Total	5583358.510	61			
Corrected Total	1442585.868	60			

a. R Squared = .457 (Adjusted R Squared = .385)

Due to the significant interaction, separate 1-way ANOVAs were performed to compare cements for both zirconia and lithium disilicate. The 1-way ANOVA for difference cements with zirconia crowns was not significant ($p=.47$).

Table 18. 1 way ANOVA for difference cements with zirconia crowns.

1-way ANOVA

Force (zirconia)

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	34267.010	3	11422.337	.879	.465
Within Groups	325019.703	25	13000.788		
Total	359286.713	28			

The 1-way ANOVA for difference cements with lithium disilicate crowns was significant ($p<.01$).

Table 19. 1 way ANOVA for difference cements with lithium disilicate crowns.

1-way ANOVA

Force (lithium disilicate)

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	617284.018	3	205761.339	12.563	.000
Within Groups	458594.447	28	16378.373		
Total	1075878.465	31			

For lithium disilicate, the Tukey post-hoc analysis determined that Luting Plus was significantly less retentive than all other materials.

Table 20. Tukey post-hoc analysis for difference cements with lithium disilicate crowns.

Tukey post-hoc

Force (lithium disilicate)

CTcement	N	Subset for alpha = 0.05	
		1	2
Luting Plus	8	53.6863	
Panavia	8		252.9375
Unicem	8		358.1937
Ultimate	8		419.3450
Sig.		1.000	.066

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 8.000.

7.3.3 Failure Mode Analysis

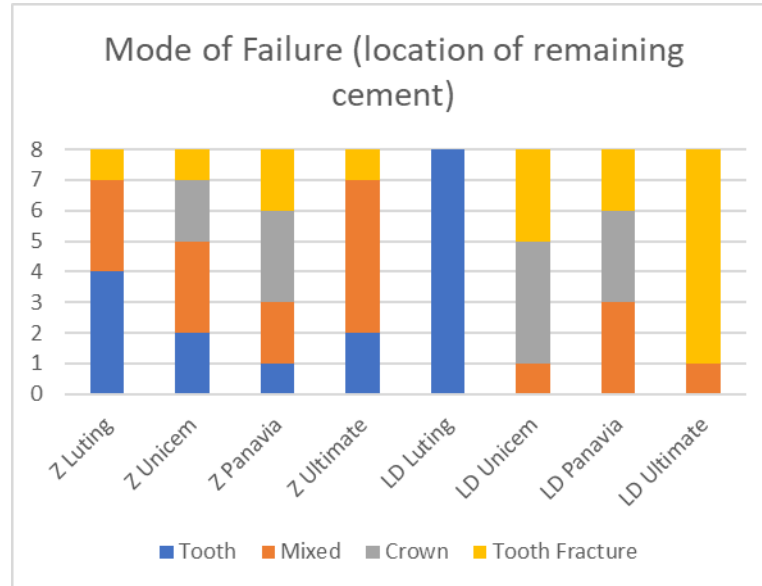


Figure 21. Failure mode analysis of different cements for bonding zirconia and e.Max crowns.

8. NULL HYPOTHESIS REJECTION

1. Significant differences were found between the retention strength of lithium disilicate crowns cemented with resin cement, RMGI cement, and GI cement albeit no difference between RMGI cement and bioactive cement. Hence, the null hypothesis that there is no difference for the retention strength of lithium disilicate crowns to prepared human teeth cemented with glass ionomer, RMGI, resin, and bioactive cements is rejected.

2. The experimental resin cement had a significantly different retention strength than two other resin cements while no difference than another three resin cements. Therefore, the null hypothesis that there is no difference for the retention strength of lithium silicate crowns to prepared human teeth cemented with a novel resin cement to other clinically successful resin cements is also rejected.

3. Overall, there is significant difference between different cements for the retention of lithium disilicate crowns, but not for that of zirconia crowns. The null hypothesis that there is no difference for the retention strength of zirconia and lithium disilicate crowns bonded with a RMGI cement, a self-adhesive resin cement, a resin cement used with a bonding agent and a novel self-adhesive resin cement following 30,000 cycles of thermocycling is also rejected.

9. DISCUSSION

The overall results of this study showed that there is significant difference between different cements for the retention of zirconia and lithium disilicate. Consequently, the null hypotheses were rejected.

In previous crown retention studies, 3-5 mm crown height preparations were used which is closer to the clinical situation. In these studies, mostly weaker cements such as zinc phosphate cement, glass ionomer cement, and RMGI cement were used.³⁰⁻⁴² It is reasonable that higher strength resin cement could cause fractures of tooth and/or ceramic specimens after crown retention test is performed. Indeed, some studies reported higher tooth fracture rates even up to 80%. In one of those studies, it was shown that tooth fracture rate would increase linearly from 20% to 80% when the failure stress raised from 3MPa to nearly 7MPa.³⁰ This finding was very consistent to our preliminary studies. In one of our preliminary studies in which all the tested cements were resin cements, the teeth were prepared to obtain a 3mm height; however, the majority of the specimens (60%) fractured during the crown retention test, specifically for specimens with the highest crown retention values. In other words, there seemed to be a maximum adhesive stress between the cement and the tooth/crown above which there would be cohesive failures within the ceramic crown or tooth. Accordingly, a reduced height around 2mm was later utilized for further projects when resin cement was used. More favorable results (less tooth fractures) were attained after modification of crown height preparations. From part 3 of our study, a total 16 out of 48 specimens were fractured for the groups of resin

cements. That is 33.33% of the specimens compared to 60% of the specimens which fractured in part 2 of the study. Nonetheless, a very high fractured rate (87.5%) was noted for the RelyX Ultimate group when cementing lithium disilicate crowns.

Different types of cement or cementation technique utilized can contribute to clinical differences in retention and ultimately the long-term success of fixed prostheses. Conventional cementation with ZPC or GI cements is generally considered weaker than RMGI or resin cements. In previous studies, GI cements typically demonstrated one-half to one-third the bond strength of resin cements.^{43, 44} In a previous study, a self-adhesive resin cement showed a higher retentive strength than crowns cemented with GI or ZPC cements.⁴⁵ This can also be confirmed from our part 1 study. From our results, one self-adhesive resin cement (Calibra Universal) was significantly more retentive than the other two RMGI cements (RelyX Luting Plus, FujiCem 2) and GI cement (Ketac Cem). One RMGI cement (RelyX Luting Plus) was significantly more retentive than GI cement (Ketac Cem), but the other one (FujiCem 2) was not.

Some in-vitro studies have also shown that adhesive resin cementation has the benefit of increasing the retention of lithium disilicate crowns⁴⁶, improving the fracture resistance of lithium disilicate crowns⁴⁷ and increasing the fatigue resistance of zirconia crowns⁴⁸. Additionally, marginal adaptation can also be improved by durable adhesion to prevent microleakage and secondary caries.⁴⁹⁻⁵² For cases in which good isolation cannot be achieved, RMGI cement is more resistant to salivary contamination and if bonding to saliva contaminated dentin, an RMGI cement is more retentive than a resin cement.⁵³

Bioactive cements were introduced claiming that these permanent cements can help prevent further secondary caries formation between the restoration and tooth margin.

However, the retentive ability of these materials has not yet well-documented. A study conducted by Jefferies et al. reported similar retentive ability compared to two currently available luting agents.⁵⁴ From our current study, a bioactive cement (Calibra Bio) displayed no difference in retention compared to two RMGI cements, but it had significantly better retentive strength than a GI cement and less retentive strength than a resin cement.

When deciding adhesive or conventional cementation for ceramics, there are several factors dentists should consider that may affect the final results which include the shade and translucence of cements, the type of ceramics (lithium disilicate or zirconia), the strength of the ceramic, the retention and resistance form⁵⁵, the location of margin⁵⁵ and so on.

For silica-based ceramics, like lithium disilicate, using adhesive cementation by resin cements is extensively recommended and is the most popular method.⁵⁵⁻⁵⁸ Bonding with resin cements can help blend the restoration's color and improve the esthetic results⁵⁹⁻⁶². Additionally, it can help prevent crack formation by infiltrating resin into the surface irregularities of a restoration's inner surface, transferring the stress from ceramic to tooth via tooth/resin cement/ceramic interface, thus preventing fracture and reinforcing the restoration.^{58, 63, 64}

On the other hand, there are some advantages of RMGI cements. It has been reported that there is no difference in retention of zirconia crowns cemented with RMGI cement and bonded with resin cement⁶⁵, no difference between fracture resistance of zirconia restorations after cementing with RMGI or resin cements⁶⁶⁻⁶⁸, no difference between fracture resistance of lithium disilicate crowns after cementing with RMGI or resin cements.^{67, 69, 70} An in vitro study also reported that there was no difference between

a RMGI cement and resin cements for retention of zirconia restorations even after thermocycling and 1 year water storage.⁴⁵ Moreover, a study conducted by Blatz et al. reported that RMGI cements were more resistant to artificial aging compared to resin cements even though they had lower bonding strength.⁷¹ RMGI cements also benefit from their ability to release fluoride and potentially antimicrobial effect than resin cements.⁷² Compared to resin cement which occasionally cause post-operative sensitivity, RMGI cement seldom have this issue.^{26, 73, 74}

Crown resistance form remains critical. In other words, if the preparation is minimally invasive or the resistance form is insufficient, such as a short crown height or tapered preparation, clinicians will be more likely to utilize adhesive cementation in order to prevent crown dislodgement.^{55, 75, 76}

In addition, another consideration for using resin cements is their poor moisture affinity. When a preparation margin is deep subgingivally and the bonding procedure may be compromised, RMGI cements which have reduced moisture sensitivity usually are the cement of choice.^{77, 78}

Part 3 of our study reported that there was no significant difference for the retention force or stress when cementing zirconia crowns between RMGI or resin cement. However, there was significant difference for the retention force or stress when cementing lithium disilicate crowns between RMGI or resin cement. Both results were consistent with current literature.^{46, 65} The low retention strength for the RMGI group when cementing lithium disilicate was further deteriorated by the absence of hydrofluoric acid applied in current study. A study conducted by Kalavacharla et al. reported that both hydrofluoric acid and silane application are required to get optimal bonding strength between lithium disilicate and resin.⁷⁹ In their study, the bond strength of resin to lithium

disilicate decreases significantly without the use of hydrofluoric acid, whether silane was applied or not. These results help explain the low retention strength of lithium disilicate cemented with RMGI cement in our current study. The mode of failure analysis also showed that the cement remained entirely on the tooth side instead of the ceramic side, indicating that all the failures were from weak bond at the ceramic interface. On the contrary, two clinical studies evaluating clinical performance of RMGI cemented to lithium disilicate restorations showed no loss of retention after a mean of 24 and 79.5 follow-up months.^{80, 81} One of these study utilized a “pre-condition oriented cementation” protocol to select suitable cases which abutment height was more than 4mm for cementing with RMGI.⁸⁰

In a systemic review from 2019, 1280 patients receiving 2436 zirconia and lithium disilicate crowns in seventeen studies were evaluated.⁸² Similar results were found comparing the clinical outcomes between adhesive bonding or conventional cementing methods for both zirconia and lithium disilicate tooth-supported single crowns. That is, conventional cementation could be an acceptable alternative to adhesive cementation which is more time consuming and technical sensitive. However, the authors emphasized that the strength of the existing evidence is weak, so long-term well-designed randomized clinical trials were required to elucidate more convincing answers. Besides, the similar survival rates measured in this study couldn't completely represent equal retentive ability of adhesive or conventional cementation.

When adhesive bonding is utilized, the choice of resin cement can be further differentiated into three types, which are total-etching resin cement, self-etching resin cement and self-adhesive resin cement. For total-etch resin cement, multiple steps are required including etching with phosphoric acid on the tooth surface, applying tooth

primer on tooth and utilization of a resin cement. Self-etching resin cement still requires tooth primer/adhesive to be applied on the tooth surface first before cementation; however, it doesn't require a separate etching step on the tooth surface. It has been shown that self-etching systems have a lower bonding strength to enamel compared to total-etching systems. The last category, self-adhesive resin cements, have gained popularity by offering dentists a high level of convenience as etchant, primer or adhesive application is not required before cementation. Due to an acidic resin monomer included, self-adhesive resin cements are capable of simultaneously demineralizing dentin and infiltrating the collagen matrix. Therefore, the dentists can benefit clinical time saving. Other advantages of self-adhesive resin cement are that some are fluoride releasing, moisture resistant and cause less post-operative sensitivity because of a preserved smear layer.⁸³⁻⁸⁵ Some studies reported that self-adhesive resin cements didn't have lower retentive strength than self-etching resin cements.⁴⁵ On the other hand, other studies showed that self-adhesive resin cements had inferior bonding strength to enamel than self-etching systems and similar or inferior bonding strength to dentin than self-etching systems.^{83, 86, 87}

In brief, even though self-adhesive resin cement is very convenient, total-etching resin cement still remains the gold standard when more retention is needed. From our study, when bonding to lithium disilicate crowns, significantly better retention stress was found for the total-etching resin cement (RelyX Ultimate) compared to one of the self-adhesive resin cements (Panavia SA Universal) yet no difference to another self-adhesive resin cement (RelyX Unicem 2). For the retention force, no significant difference could be found between these three cements. Aside from the difference in retention strength, the mode of failure was also different for the Rely X Ultimate cement. In this group, 7 out of 8 teeth fractured during debonding. This observation implies that value recorded

for retention strength was in fact the cohesive strength of the tooth, and the true retention strength would be higher. When bonding to zirconia crowns, no significant difference was found between two self-adhesive resin cements (RelyX Unicem 2, Panavia SA Universal) and one total-etching resin cements (RelyX Ultimate).

Panavia SA Universal (Kuraray Co.) is a novel self-adhesive resin cement that was made commercially available in late 2019. Unlike most mainstream resin cements, this cement incorporated not only 10-MDP monomer (for bonding to zirconia) but also silane to foster the bond with lithium disilicate. Due to the 10-MDP monomer included, current resin cements usually have good bonding ability to zirconia prosthesis once sandblasting is performed. When bonding to lithium disilicate prostheses, however, a separate ceramic primer must be applied to the crown surfaces as a mandatory procedure. By including silane in the cement, there is no need to further apply silane before cementation, which can contribute to an easier way to cement lithium disilicate prostheses. Due to these advantages, the novel Panavia SA Universal could provide extreme convenience and efficiency to cement nearly all kinds of prostheses. The silane agent included in this cement has a long carbon-chain. This modification to the silane molecule was performed in order to allow sufficient silane monomer to be exposed at the interface of the cement and the lithium disilicate surface. Currently there is no data regarding the performance of this newly-developed products. From our study results, the Panavia SA Universal without further silane application could achieve statistically similar retention strength compared to another self-adhesive resin cement (RelyX Unicem 2) in which silane was applied, albeit the retention strength was numerically lower for cementing lithium disilicate crowns.

10. LIMITATIONS

1. When crown retention strength is high for stronger resin cements, there is a limitation to test because of higher possibility of crown fractures.
2. Crowns in the second study fractured during crown retention testing, so the values obtained for these cements may represent cohesive strength of substrate rather than bond strength.
3. To reduce crown fractures during the third test, short and tapered crown preparation designs were utilized, however, these preparations might not completely reflect the true clinical scenario.
4. Crowns were thermocycled between 5 and 55°C to artificially age the bond. Load cycling the crowns would have also been useful, however, the technical limitation of load cycling is the occlusal surface of the specimens would not allow loading.
5. The crowns were debonded by applying a tensile load through the long axis of the tooth preparation. This debonding force may not accurately represent clinical debonding, however, this debonding strategy allowed standardization of testing.

11. CONCLUSION

Within the limitations of the present study, the following conclusions can be drawn:

1. The self-adhesive resin cement demonstrated greater retention compared to bioactive, GI and RMGI cements.
2. The “bioactive” calcium aluminate cement (Calibra Bio) and one of the RMGI cements (Rely X Luting Plus) achieved greater retention than the glass ionomer cement (Keta Cem).
3. The other RMGI cement (FujiCEM 2) performed similarly as the glass ionomer cement (Keta Cem).
4. The experimental resin cement in study 2 had a significantly greater crown retention strength than NX3 and Panavia V5. It had a statistically equivalent crown retention strength as Link Force, Calibra Ceram, and Variolink Esthetic.
5. The large number of tooth fractures during crown retention test can be credited to the high retention strength achieved between lithium disilicate and resin cements AND between tooth structure and resin cements used with tooth primers.
6. Teeth fracture rate can be decreased if a shorter (2mm) and more tapered (23 degree) tooth preparation design was utilized compared to a longer (3mm) and

less tapered design (16 degree) for crown retention tests when resin cements were used.

7. No significant difference can be found between RMGI and resin cements when cementing to zirconia crowns. However, significant better retention strength can be found for resin cement compared to RMGI cement when cementing to lithium disilicate crowns.
8. No significant difference can be found between self-adhesive and total-etching resin cements when cementing to zirconia crowns. However, significant better retention stress (MPa) was found for a total-etching resin cement (RelyX Ultimate) compared to one self-adhesive resin cement (Panavia SA Universal) yet no difference for another self-adhesive resin cement (RelyX Unicem 2) when bonding lithium disilicate crowns. For the retention force (N), there was no significant difference between these three cements.
9. The Panavia SA Universal without further silane application could achieve statistically similar retention strength compared to another self-adhesive resin cement (RelyX Unicem 2) in which silane was applied. However, it should be emphasized that higher retention strength still could be achieved for the resin cement used with adhesive (RelyX Ultimate) used with silane compared to Panavia SA Universal without silane when cementing lithium disilicate crowns.

12. FUTURE DIRECTIONS

1. Comparing the retention strength for the lithium disilicate crowns to human teeth cemented with RMGI and resin cements when a silane coupling agent is used or not.
2. Using molar teeth for specimens while a standardized tooth preparation protocol is still utilized.
3. Applying better designs for preparation such as increasing crown height while preventing specimen fracture rate.
4. Testing the retention strength on dental implant abutments instead of human teeth with different cements.
5. Testing and comparing the retention strength of different provisional cements cemented on human teeth.
6. Comparing the retention strength with newer ceramics or newer kinds of dental luting agents.

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APPENDIX A

INSTITUTIONAL REVIEW BOARD APPROVAL

**UAB THE UNIVERSITY OF
ALABAMA AT BIRMINGHAM**
Office of the Institutional Review Board for Human Use

470 Administration Building
701 20th Street South
Birmingham, AL 35294-0104
205.934.3789 | Fax 205.934.1301 | irb@uab.edu

NHSR DETERMINATION

TO: Lawson, Nathaniel

FROM: University of Alabama at Birmingham Institutional Review Board
Federalwide Assurance Number FWA00005960
IORG Registration # IRB00000196 (IRB 01)
IORG Registration # IRB00000726 (IRB 02)

DATE 15-Oct-2018

RE: IRB-300002419
Retention of Lithium Disilicate Copings Luted with New Dental Adhesive

The Office of the IRB has reviewed your Application for Not Human Subjects Research Designation for the above referenced project.

The reviewer has determined this project is not subject to FDA regulations and is not Human Subjects Research. Note that any changes to the project should be resubmitted to the Office of the IRB for determination.

if you have questions or concerns, please contact the Office of the IRB at 205-934-3789.