THE INFLUENCE OF RESIN CEMENT THICKNESSES ON SHEAR BOND STRENGTH OF ZIRCONIA TREATED WITH A UNIVERSAL ADHESIVE

BY

MR. APICHAI MANEENACARITH

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER SCIENCE (RESTORATIVE AND ESTHETIC DENTISTRY)

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ENTITLED

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was approved as partial fulfillment of the requirements for the degree of Master of Science program in dentistry (Major in Restorative and Esthetic Dentistry)
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Thesis Title	THE INFLUENCE OF RESIN CEMENT THICKNESSES ON SHEAR BOND STRENGTH OF ZIRCONIA TREATED WITH A UNIVERSAL ADHESIVE
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ABSTRACT

Objective: The study examined the influence of resin cement thicknesses on shear bond strength of zirconia treated with a universal adhesive.

Materials and methods: Forty zirconia specimens (6 mm diameter, 4 mm thickness) were prepared, polished, surface treated with universal adhesive, and divided into 4 groups according to resin thicknesses (50, 80, 160 and 240 µm). All samples were stored in 37°C distilled water for 24 hours. The shear bond strength was performed using a universal testing machine at a crosshead speed of 0.5 mm/min. The results were statistically analyzed by one-way ANOVA and Tukey's multiple comparison test at a 95% confidence level. The failure modes were analyzed using stereomicroscope. Samples from each group were randomized and further analyzed under scanning electron microscope with the magnification of x2000

Result: The shear bond strength for each group 1, group 2, group 3 and group 4 were 30.28±3.09, 26.86±2.21, 25.98±2.96, 18.22±1.71, respectively and Group1 showed significantly the highest bond strength than the other groups ($P < 0.05$). There were no significant differences between Group2 and 3 ($P > 0.05$). Group 4 showed significantly the lowest bond strength than the other groups ($P < 0.05$). The adhesive failure and mixed failure were found in all groups.

Conclusions: Resin cement thicknesses had the influence on zirconia shear bond strength treated with a universal adhesive. The thinner resin cement showed higher shear bond strength than the thicker resin cement thicknesses.

Keywords: Resin cement, Shear bond strength, Universal adhesive, Zirconia and Resin cement thickness

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Apichai Maneenacarith

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LIST OF ABBREVIATIONS

Symbols/Abbreviations	Terms
MDP	Methacryloyloxydecyl dihydrogen phosphate
GPT-9	Glossary of prosthodontics
ADA	American Dental Association
TZP	Tetragonal zirconia polycrystal
SIE	Selective infiltration of etching technique
MPa	Megapascal
W	Watt
ToF-SIMS	Time-of-Flight Secondary Ion Mass Spectrometry
NMR	Nuclear magnetic resonance
Wt	Weight
CAD	Computer aided design technology
ISO	International standard organization
PVC	Polyvinyl Chloride
SD	Standard deviation
SEM	Scanning Electron Microscope
µm	Micrometer or micron

CHAPTER 1
INTRODUCTION

1.1 Introduction

Ceramics have been widely used in restorative dentistry for many years. In the past, glass ceramics were chosen as indirect restorations because of its excellent translucency and esthetics [1]. Due to its brittle nature, these glass ceramics can cause material chipping or fracture which leads to restorative failure. Bonding of glass ceramics can be created using hydrofluoric acid and followed by silane application to obtain a suitable adhesion according to many recommendations [2]. In the present, zirconia gained its popularity as an indirect restoration since there have been improvement in its biocompatibility, esthetic and mechanical properties [3].

Zirconia is a polycrystalline ceramic without a glassy phase like other glass ceramics. To gain a suitable retention, zirconia has some inherent problems. Surface treatment of etched zirconia with hydrofluoric acid does not adequately roughen the surface for purpose of retention, while grinding zirconia to create surface roughness was often used as an option to improve its mechanical bonding [4]. Retention of zirconia restoration can be gained by both mechanical and chemical procedures. In the past, grinding methods have been investigated for gaining mechanical retention as they were easily assessible. However, some studies have been reported that grinded surfaces create microcracks which compromised zirconia mechanical properties [5, 6]. Therefore, an alternative method has been suggested to create surface irregularity for improvement of zirconia bond. The irregular surface can promote mechanical interlocking between cement and the zirconia. A well-recognized protocol for increasing mechanical retention is grit blasting or sandblasting [7]. Sandblasting is a technique where aluminum oxide particle is released to attack the surface of zirconia creating roughness on the surfaces. Surface irregularity is also known to increase surface energy and wettability of the zirconia restoration [8]. Many studies had suggested that sandblasting can be a method that gains mechanical retention in zirconia restoration as zirconia cannot be etched by hydrofluoric acid in normal condition [7, 9, 10]. However, a chairside sandblasting device must be invested by the dental practitioner, and thus, increasing cost and chair time.

To further increase the longevity of the bonded zirconia restoration, chemical retention has been recommended [7, 11]. The traditional bonding methods have been widely used and considered an effective bonding procedure on both zirconium restoration and the tooth abutment, in which the application of primer and bonding agents are required before cementation [7]. These primers and bonding agents contain an essential functional monomer which will be bonded to the zirconia's surfaces. This traditional bonding requires multiple steps for surface treatment. Nowadays, a novel adhesive model has been released into the market which is known as "Universal adhesive" or "Multimode adhesive". It aims to reduce clinical steps and errors [12]. Universal adhesive tries to incorporate potential chemical monomers into a single bottle which is designed to bond with both direct and indirect restorations including ceramics, composites and metals [13, 14]. This provides dentists with more options when choosing an optimal protocol for bonding with different prepared cavities. Many studies have reported that the use of universal adhesive can improve the bond strength of the zirconia [14-16]. The study of Santos R. et al in 2019 has shown that the use of universal adhesive may provide an adequate shear bond strength of zirconia for clinical use [14]. Kim et al. in 2015 found that the Universal adhesive exhibits significantly higher bond strength in comparison with the primers containing conventional MDP (Methacryloyloxydecyl dihydrogen phosphate) [17]. Moreover, a recent study has claimed that universal adhesive may provide a sufficient shear bond strength on zirconia as an alternative option to traditional bonding [18].

The functional monomer that provides chemical retention for zirconia is typically 10-MDP which is an acidic functional monomer containing phosphate group. It is capable to create chemical bond with the metal oxide surface [19]. Zirconia surface is covered with an oxide layer which makes it similar to a metal surface and researches have proven successfully in regards to the application of 10-MDP to increase the bond strength of zirconia [20]. However, the problem with MDP is that it is susceptible to hydrolysis. This makes the interface become prone to leakage [21]. Even so, 10-MDP is still an important aspect in most dental bonding agents [19, 20, 22].

Dental cement plays an important role in filling up the internal gap of dental ceramic restoration with the tooth abutment. Various dental resin cements are available in the market which provide options for luting dental restorations. Resin cements contain various compositions according to different manufacturers in regards to modes of cure and types of surface treatment. These variations can differ the dental cement properties such as thickness, viscosity and even strength [23]. According to Grajower and Lewinstein in 1983, the cement space of a crown should be set to be at least 50 µm where the space of 30 µm would be reserved for the cement thickness and 20 µm would serve as a potential distortion during the restoration fabrication [24]. A study of McLean and Fraunhofer in 1971 reported that 100-120 µm cement thickness provided an acceptable long term clinical prognosis for the indirect restoration [25]. The recent study from Taha and Ibrahim in 2019 suggested that the use of 80 µm spacer thickness resulted in significantly higher retention than 100 µm and 120 µm in zirconia restoration [26]. In the present days, researchers have investigated on the effect of resin cement thickness that affected mechanical properties of bonded restoration such as bond strengths, fracture toughness and color translucency [27-29]. A poor internal adaptation can lead to an increase in resin cement thickness which may affect the bond durability of the restoration [30]. This suggested that variations of cement thickness may have an effect on the bond strength of zirconium restoration. Many experiments have been performed to measure the resin-zirconia bond strength under different conditions such as surface treatments, resin cements, and the use of primers with other factors [10, 14, 20, 31]. Until todays, there has been no study that evaluates the resin-zirconia bond strength under conditions differing in the thicknesses of resin cement together with universal adhesives. Therefore, the purpose of the present study was to evaluate the influence of cement thickness on shear bond strength of surface treated zirconia with a universal adhesive at the minimum cement thickness of 50 µm, recommended thickness 80 µm and the highest cement thickness 240 µm.

CHAPTER 2
REVIEW OF LITERATURE

2.1 Ceramics

Ceramics are composed of one or more metallic with a non-metallic element (GPT-9). These ceramics have been a material of choice for esthetic dentistry for many years. Originally, it is used as a veneering material by layering over the metal substructure to enhance esthetics. Over the years, ceramics have been improved on its strengths, esthetics and fabrication methods which provide options for clinicians [32]. Ceramics can be classified into 3 major categories according to its composition as follows: glass-matrix ceramics, resin-matrix ceramics and polycrystalline ceramics [33]. The first traditional glass-matrix ceramic is feldspathic porcelain. Feldspathic porcelain dominantly composed of feldspar, kaolin and quartz. It has been used as a veneering material due to its glassy dominant composition causing the material to be highly translucent; however the major disadvantage is that the material can be easily fractured [33]. In order to increase the material strength, manufacturers try to synthesize the materials with different leucite crystals. The synthetic leucite crystal provides an increase in physical properties. It made the subgroup material called leucite reinforced and lithium disilicate [1, 33, 34]. These leucite crystals contain a crystal which has refractive index similar to dentin. The next subgroup is glass-infiltrated ceramics, a porous ceramic, which is infiltrated with lanthanum glasses causing an increase in physical properties after firing. The strength and properties of glass infiltrated ceramics differ due to different material compositions [1]. However, the use of glass infiltrated ceramics has decreased as the popularity of lithium disilicate and zirconia increased. The second category comprises of an organic matrix filled with ceramic particles and is called resin-matrix ceramics. ADA has defined most dental ceramic as "pressed, fired, polished or milled materials containing predominantly inorganic refractory compounds – including porcelains, glasses, ceramics, and glass-ceramics". The resin-matrix ceramics predominantly (>50% by weight) consist of refractory inorganic compound. This resin matrix ceramic was made to create a close elastic modulus to dentin and provides an option to repair with resin composites [33, 35]. The third category is polycrystalline ceramic, ie. zirconia, which is a crystalline structure without the glassy phase. Although the improved physical properties of this ceramic come from the densely packed crystalline, it comes with a decrease translucency. The polycrystalline ceramic does not contain glassy phase which makes it hardly able to increase the surface roughness by etching with hydrofluoric acids as it requires long etching time or a higher temperature [31, 36].

2.2 Zirconia

Zirconia was first used as a biomaterial in total hip replacement [3]. Over the years, this material has been improved with combination of different stabilizing oxides. In the present day, the combination of yttria oxides with zirconia is commonly used. This is also known as Tetragonal Zirconia Polycrystal (TZP). Zirconia is a polymorphic material which changes its structure without changing its chemical properties [3]. Zirconia exists in three forms: monoclinic at low temperatures, tetragonal above 1170 °C and cubic above 2370 °C. A well-known characteristic is that it changes in the crystal structure from tetragonal to monoclinic which results in a volumetric increase by 3-4%. By adding 3 mol of yttrium (3Y-TZP), it allows zirconia to be partially stabilized in tetragonal form at a room temperature [37]. The cubic and tetragonal phases are kept while phase transformation from tetragonal to monoclinic only occurs when external stress is applied. This process is known as transformation toughening which provides zirconia with a volume expansion of approximately 4%. Furthermore, the compressive stress that occurs at the tips of the cracks also undergoes phase transformation which prevents crack propagation [38]. In dentistry, zirconia is known for its high flexural strength around 600-1400 MPa [32]. A monolithic zirconia is mostly used as a crown substructure or used in an area where less esthetic is required due to its lack of translucency [35]. To tackle this problem, a translucent zirconia is developed, by addition of more yttria and cubic phases, improving its esthetic properties [39]. However, its flexural strength and toughness have reduced as they undergo less stress-induced transformation [40]. Even though zirconia has been studied over the years, many researchers still try to invent a better protocol to improve adhesion for this material. The generally accepted standard practice is to create both mechanical and chemical retention for zirconia [7, 16, 20, 41].

2.2.1 Mechanical retention for zirconia

Since zirconia contains a pure crystal structure without any glassy phases, this makes it difficult to be etched with hydrofluoric acid, unliked the other glass ceramics. Other alternative methods have been tested to gain mechanical retention such as: surface abrasion [4, 5, 41], selective infiltration of etching technique (SIE) [42], hot chemical etching solution [42] and laser treatment [43].

For examples, the first suggested method is grinding which is easy to apply and to get access in dental environment. However, compared with sandblasting [5], the use of diamond burs showed for grinding several disadvantages such as higher material removal, increased in stress and temperatures resulting in lower mean strengths. The high stress from grinding could induced surface microcracks which may decrease zirconia physical properties [5]. Therefore, sandblasting is generally recommended [9]. Sandblasting is a method which aluminum oxide particles are used at a various size to create surface irregularities on the zirconia surface. Various factors of sandblasting can affect its efficacy such as: time, pressure and size of alumina particles. The study from Su et al. in 2015 suggested that sandblasting with the alumina at 0.2 MPa for 20 seconds with 110 μm particles is recommended to improve the bonding of zirconia [9].

The second method in gaining mechanical retention is selective infiltration etching technique (SIE) [42]. It is the process which heat is used to increase pre-stressed grain boundary of zirconia by decreasing melting glass infiltant, After that, zirconia is immersed in hydrofluoric acid creating nano-porosity in the grain boundary. This allows low viscosity resinous materials to penetrate and to create mechanical interlocking after polymerization. From the study of Casucci in 2009, SIE technique creates a significant surface roughness which is higher than sandblasting itself [42].

The third method is hot chemical etching solution where an experimental hot etching solution was heated up to 100 and applied for 10-60 min according to the protocol which was proposed by Ferrari in 1989 [44]. From the study of Casucci in 2009, the surface roughness of zirconia was significantly increased compared to the other methods ie airborne particle abrasion, hydrofluoric acid and, moreover, the roughness was increased as the application duration increased. Even though this method can produced the highest surface roughness, it might not be applicable in clinical dental environment [42].

The fourth method is the laser treatment that can be used to create surface roughness on zirconia. From the study of Ural in 2010, A 3W CO_2 laser surface treatment had achieved the highest shear bond strength compared to the use of hydrofluoric acid and sandblasting. However, main concern with the use of laser surface treatment is pulp damage when the treatment is carried at dentin surface [43].

2.2.2 Chemical retention for zirconia

There are several ways to gain chemical retention in zirconia restoration such as application of phosphate monomer and silane coupling agents. Since zirconia does not contain silica, many studies involve a tribochemical silica coating together with the application of silane coupling agent to assist the non-silica-based ceramics [8, 20, 45, 46]. Silane coupling agent in dentistry is also known as a product which contain trialkoxysilane and in order for silane to function properly, it needs to be hydrolyzed at a suitable pH. Silane contains two groups of side chains. One end is an organofunctional group which can be polymerized with an organic resin. The other side chain is a hydrolysable alkoxyl group which can be reacted with silica. Two important steps in bond formation using silane coupling agents are 1) silane and substrate bond formation and 2) resin and silane bond formation [47]. Silica coated air abrasion or tribochemical silica coating was used to enhance chemical retention in zirconium restoration [20]. Alumina oxide particles will be blasted with silica creating surface roughness and then covered the abraded surface with silicon dioxide, resulted in both silanization and micromechanical retention after silane application. A study of Pilo et al. in 2018 reported that zirconia surface with tribochemical silica coating was the most reliable treatment [46]. The study of Xie in 2016 also reported that using tribochemical blasting can produce higher bond strength than non-blasting [21].

10-MDP is an important substance which significantly promote chemical retention of zirconia and resin cement. 10-MDP is a bifunctional molecule containing phosphate group that is capable of bonding with oxide layer on zirconia surface. Many studies have evaluated 10-MDP contained in cements or primers used with zirconia bonding and shown that 10-MDP provided a durable bonding between zirconia and resin composites [8, 10, 19, 20, 22]. Erdem et al. in 2014 reported that the use of the phosphate monomer containing resin cement together with alumina blasting or tribochemical silane application on zirconia surfaces produced a higher bond strength [48]. Another study from Souza et al. in 2014 examined non-airborne particles abraded zirconia surface with the use of different MDP-based materials. This study found that the use of MDP-based adhesive systems significantly increased the bond strength of zirconia when used with non-airborne particles abraded surface [11]. In addition, the study of Kitayama et al. in 2010 stated that resin cement containing 10-MDP could create an effective bonding to a zirconia even though primer was not used. As 10-MDP have an ability to bond with the oxide layer which is similar to metal oxides [49]. From Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) study of Lima in 2019, phosphate, oxygen and zirconium bond showed the greatest bond strength. Moreover, the microshear bond strength did not decrease after 8 months of storage in distilled water when MDP-based primer was applied after zirconia was abraded with airborne-particles [22]. Furthermore, a Nuclear Magnetic Resonance (NMR) analysis by Nagaoka et al in 2017, confirmed that a strong bond interaction between 10-MDP and zirconia was formed where phosphate group from 10-MDP interacted with the oxide group of zirconia [19]. In the present days, 10-MDP is widely used in everyday dental bonding system and even dental adhesive.

2.3 Dental adhesive

Dental adhesive is a crucial step in ensuring durability of bonded restoration. Bonding agents can be categorized according to modes of surface treatment on the tooth abutment and restoration [50]. The traditional bonding system was first introduced where multiple steps of the treatment must be prepared before applying adhesive. Many studies have reported on the traditional bonding system which have been proven to be an effective bonding agent [7, 18, 51]. However, the problem with this bonding is that it requires multiple steps which are technical sensitive [50].

In the present days, a new adhesive has been released to reduce clinical steps known as universal adhesive or single step adhesive. This adhesive contains various chemical substances which promote adhesion to both tooth structures and almost all indirect restorations [14, 16, 52, 53]. Universal adhesive system or also known as multimode adhesive system has been designed to bond with different materials. Lümkemann et al. in 2019 discussed that the recent universal adhesive contained MDP and other different functional monomers have different effects. The 10% wt of 10-MDP concentration had the most effect on the shear bond strength of zirconia and resin composite. This study concluded that universal adhesive can promote resin bonding with zirconia [15]. Another study from Al Jeaidi in 2017 supported that the use of universal adhesive promotes the bond strength of zirconia posted cementation and claimed that the effect of the functional monomer was to increase shear bond strength of zirconia, since 10-MDP forms a covalent bond between oxygen, phosphorus and zirconia [16]. Santos et al. in 2019 mentioned that universal adhesive application on zirconia could create a good bond strength in the range of 15 to 25 MPa which is considered a good clinical parameter [54]. Therefore, universal adhesive can be used with zirconia to simplify clinical steps. Recently, a study of Klaisiri et al in 2019 concluded that universal adhesive can be an alternative material to replace zirconia primer for the surface treatment [18]. In addition, Kim et al. in 2015 also found that the universal adhesive exhibits significantly higher bond strength in comparison to the primers containing conventional MDP [17]. So far, research information about universal bonding suggested that universal adhesive may be an alternate method to traditional bonding to gain bond strength in the indirect restoration.

2.4 Dental cement thickness

An International Standard for polymer-based filling, restorative and luting materials mixing ratio of base and catalyst ISO 4049:2019 set the critical thickness of the cement at 50 µm. However, some resin cements cannot form the thickness less than 50 µm due to different factors such as filler addition, resin consistency and viscosity [29]. Therefore, cement thickness from different manufacturers can be varied. The film thickness of resin cement available in the market range from 9.4 to 34.4 µm [55] while Multilink N resin cement film thickness is less than 20 µm according to technical data from the manufacturer. Furthermore, the applications of die spacer can affect the cement thickness. The die spacer thickness for zirconia restoration is virtually set by the computer-aided design (CAD) technology [56]. A study of Moldovan et al. in 2011 reported that the average internal gap obtained in zirconia coping produced by CAD/Cercon was 100-130 µm and CAD/Cerec was 60-70 µm, while the die spacer thickness values were set at 10-20 µm and 100 µm, respectively [57].The die spacer provides an increase in space for the cement between the prepared abutment surface and intaglio surface of the crown. This provides a reduction in stress formation during the cementation which results in better fitting and retention of the definitive restoration [58]. However, an over application of die spacer can cause an increase in resin cement thickness. From the study of Asbia et al. in 2015, it was reported that cement thicknesses can have influenced on material properties [23]. A study from Cunali et al. in 2017 reported that the marginal and internal gap of zirconia material can range from 60 (68.73±8.86) to 240 (187.35±53.89) µm [59]. According to Grajower and Lewinstein in 1983, the cement space of a crown should be set to be at least 50 µm where the space of 30 µm would be reserved for the cement thickness and 20 µm would serve as a potential distortion during the restoration fabrication [24]. The recent study from Taha and Ibrahim in 2019 suggested that the use of 80 µm spacer thickness resulted in significantly higher retention than 100 µm and 120 µm in zirconia [26]. From Leevailoj and Karntiang in 2014, the thickness of resin cement affected the fracture resistance of enamel-bonded ceramic. The thicker cement thickness causes a decrease in mean fracture load when bonded with 1 mm ceramic thickness [28]. Another study from Tuntiprawon and Wilson in 1995 showed a similar trend of cement thickness and concluded that thicker zinc phosphate cement led to the lower fracture strength of a porcelain jacket crown. A possible explanation may be due to the different physical properties of the used cement materials and the greater cement thickness allowed the porcelain to deform and therefore less force was needed to achieve the fracture strain of the porcelain [30].

On the other hand, the research of Lee et al. in 2011 reported that the different resin-cement thicknesses had no significant effect on the bond strength between the resin cement and zirconia. It was postulated that the thick cement may yield in significantly greater bond strength than the thin cement [60]. Furthermore, Urapepon and Luesak et al. in 2014 also supported that various cement spaces between 50 and 150 µm did not significantly affect the shear bond strength [29].

From the above studies, the information regarding the cement thickness that affect the shear bond strength is still controversy. So far, there is no any investigation made on the effect of the cement thickness on the shear bond strength of zirconia when bonded with universal adhesive. Therefore, the purpose of the present study was to investigate the influence of cement thickness on the shear bond strength of zirconia when bonded with a universal adhesive.

2.5 Bond strength test

Analysis of bond strength can be divided into static and dynamic tests [61]. Static test can be carried out in either macro- or micro bond test in a fixed specimen position, depending on the bonding area of the specimen. The macro-bond strength test is carried out when a bond area is larger than 3 mm^2. The specimen can be measured using either 'shear' or 'tensile' test, while in the micro-bond test, the bond area tested is much smaller, approximately 2 mm^2 or less. Dynamic test is analyzed when the specimen is in dynamic state [62]. The bond strength of the experiment can be calculated by maximum force applied and divided by the bonding area. A standardized test must be performed according to International Standards Organization (ISO) Technical Specification No. 11405 guidance. This specification provides a guideline on the material selection, storage, handling and specific method to perform a quality testing on the specimen.

Macro bond strength test can also be classified into shear and tensile [63]. Macro-shear bond strength test is used to test the new adhesive bonding effectiveness. The test of shear bond strength is carried out when two materials are connected with adhesive and then the shear force is loaded until failure. The shear bond is calculated by the highest stress where a material can withstand before failure. This test is commonly used as it is the easiest and fastest method [64].

The Macro-tensile Bond Strength test is less commonly used. Macro tensile bond strength test is performed when the load is applied on either side of the specimens. The specimen can be gripped on a testing machine using either passive or active method. An active grip requires mechanical gripping aid from a device while a passive grip does not require any mechanical grippings and the specimen is placed directly on the testing device [65]. Macro tensile bond strength is normally used to test the bond strength of cementation of hard materials such as ceramics and metal alloys [62].

Micro-shear bond strength test involves a bonding area of ≤ 3 mm^2 [66]. This allows specimens to be carried out in a small area. Armstrong et al. in 2010 discussed the findings based on the finite element analysis and failure mode analysis of macro shear bond strength test and concluded that it also appears to be true in micro shear bond strength test [65]. The micro-shear test methods are commonly tested in glass ionomers [67]. From the study of Placido et al. in 2007, it was reported that the shear bond strength test are performed due to its simple process compared to tensile bond strength test [68].

In micro-tensile bond strength test, a specimen will be bonded to a flat surface of the tooth. After curing and storage, the specimen will be prepared using two methods which are trimming and non-trimming technique. A trimming technique is where a specimen will be trimmed into an hourglass shaped with a rectangular cross section. During trimming, careful action must be performed as over trimming can provide extra stress on the bonding interface and create an origin for crack propagation [69]. Micro tensile test can be an option for evaluating an adhesive interface [70]. However, this test is harder to perform as they are technique sensitive due to specimen placement which may alter the result from the fracture load distribution. Furthermore, dehydration of the smaller specimens may occur due to further specimen processing are required after the bonding procedure [68, 71].

The research question for the present study was to determine whether the thickness of resin cement has an effect on the shear bond strength of zirconia restoration when treated with a universal adhesive.

CHAPTER 3
RESEARCH METHODOLOGY

3.1 Research Gap

The variation of dental cement spaces in zirconia restoration can vary upon different dental practitioner preparations. Dental cement space can be increased to compensate the area where undercut may be found. This may raise concerns for shear bond strength of the material when it is bonded with universal adhesives. Hence, the cement thickness must be investigated whether it would affect the strength of restoration.

3.2 Objectives

The objective was to investigate the influence of different dental cement thicknesses on the shear bond strengths of zirconia using a universal adhesive.

3.3 Hypotheses

Null hypothesis: The shear bond strengths of zirconia bonded with a universal adhesive were not different at resin cement thickness of 50, 80, 160 and 240 μm.

Alternative hypothesis: The shear bond strengths of zirconia bonded with universal adhesives were different at resin cement thickness of 50, 80, 160 and 240 μm.

3.4 Study design

Figure 3-1 Study design

The diagram (Figure 3-1) shows an experimental design in the present study. The experiment was conducted at medicinal extract and biomaterial research laboratory at Piyachart 2 building Thammasat University (Rangsit campus).

Independent variables were sandblasted zirconia disc, adhesives for surface treatment and resin cement thickness.

Dependent variables were shear bond strength and mode of failure in zirconia bonded with the resin cement

3.5 Sample size calculation

The sample size was calculated by using G*Power 3.1 Software (University of Dusseldorf, Germany) and set significance level at 0.05, power at 0.99 based on the results of a previous study [18]. The results indicated that the sample size of 10 specimens used in each test gave power >0.99 at $\alpha = 0.05$. The pilot study was performed and the calculation of effect size was made. The result from the pilot studies indicated that the sample size of 10 specimens used in each test gave power >0.99 at $\alpha = 0.05$

3.6 Materials preparation

3.6.1Preparation of zirconia specimens

The forty fully sinter zirconia specimens (6.0 mm diameter and 4.0 mm thickness) were prepared from a zirconia block (Ceramill Zolid HT+PS, Amann Girrbach, AG, Koblach, Austria) and sintered followed the manufacturer's instruction. The specimens were embedded in polyvinyl chloride (PVC) tube, and filled with dental gypsum type 4. All specimens were polished with 600 grit silicon carbide paper (3M Wetordry abrasive sheet, 3M Minnesota, USA) with 2kg/cm^2 force, 100 round/minute for 2 minutes under running water using automatic polishing machine (Tegramin-25, Struers. Inc, Cleveland, USA) and then sandblasted (A10723 Base 3, Dental Vision Co. Ltd, Bangkok, Thailand) with 50 µm alumina for 15 seconds under 3.8 bar pressure and a 10 mm distance [41]. The specimens were rinsed off using running water and cleaned for 15 minutes in distilled water using ultrasonic cleaner (WUC-D22H, DAIHAN-brand Analog Ultrasonic Cleaners, DKSH Singapore Pte LTD., Singapore). The polished specimen was shown on the left side of figure 3-2.

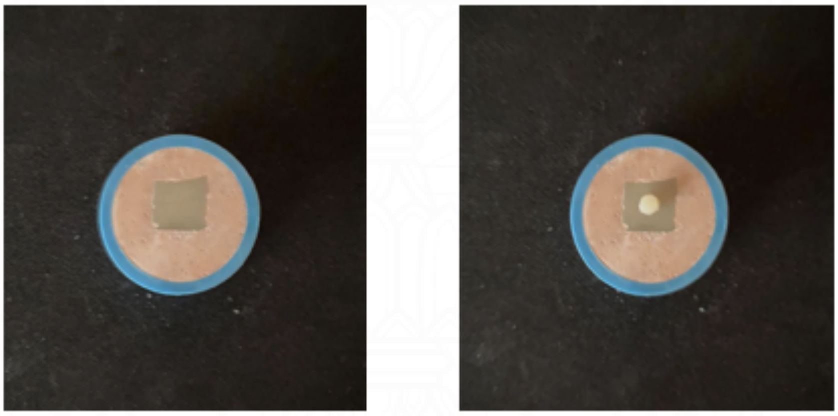

Figure 3-2 Zirconia specimen preparation

All specimens were surface treated with a universal adhesive (Clearfill Universal bond and Clearfill DC activator, Kuraray Noritake Dental Inc, Tokyo, Japan) and divided into 4 groups (n = 10 each) according to resin cement thicknesses; Group 1: 50 µm, Group 2: 80 µm, Group 3: 160 µm, and Group 4: 240 µm. Table 3-1 showed materials used in the present study.

A one-sided tape (PPM Sticky tape, PPM industries S.p.A., Brembate Sopra, Italy) with the different thicknesses 50, 80, 160 and 240 µm were cut with a dimension of 10 x 10 mm and a hole was made with the diameter of 2 mm as bonded area. One side of the tape was cut until it reaches the hole made to ease the removal of tape after the bonding procedure. A one-sided tape was adapted on the top of the zirconia surface.

Table 3-1 Chemical compositions of materials used in this study

Material name	Compositions
Ceramill Zolid HT+PS (Lot No.1905000)	$ZrO_2 + HfO_2 + Y_2O_3$: ≥ 99.0 Y_2O_3: 6,0 - 7,0 HfO_2: ≤ 5 Al_2O_3: ≤ 0.5 Other oxides: ≤ 1
Clearfill universal bond (Lot No. B10044)	Bisphenol A diglycidylmethacrylate 15-35% 2-hydroxyethyl methacrylate 10-35% Ethanol <20% Other ingredients: 10-Methacryloyloxydecyl dihydrogen phosphate Hydrophilic aliphatic dimethacrylate Colloidal silica dl-Camphorquinone Silane coupling agent Accelerators Initiators Water
Clearfill dual cure activator (Lot No. CH0009)	Ethanol > 90% Other ingredients: Catalyst Accelerator
Multilink N automix (Lot No. X51463)	Bis-GMA, ethanol, 2-hydroxyethyl methacrylate, phosphonic acid acrylate, diphenyl (2,4,6- trimethylbenzoyl)phosphine oxide, potassium fluoride

3.6.2 Application of Clearfil Universal Bond+ Clearfil DC activator

A microbrush soaked in an adhesive containing a mixture of 1 drop of clearfil universal bond and 1 drop of clearfil DC activator was applied on zirconia surface only 1 time and a new microbrush was used to clean up the excess adhesive inside the one-sided tape. A water oil free triple syringe was blown using a force of 40-50 pound/cm^2 with a distance of 10 mm until the solvent in adhesive is totally dry. To evaporate the solvent from the zirconia surface, the dry air was blown until there was no movement of the liquid and the shiny zirconia was presented. The samples were covered with a dark container to protect any contaminations before the next steps.

3.6.3 Cementation with dual cure resin cement

The resin composite rods (CeramX spheretec 1 shade A3.5, Ivoclar vivadent AG, Schaan, Liechtenstein) were prepared using a silicone mold (Honigum putty, DMG GmbH, Hamburg, Germany) with the 3 mm diameter and 2 mm height. The resin composite rods were light cured for 40 seconds using the light curing unit (Demi Plus, SDS Kerr, Middletin, WI, USA) and was held perpendicular and closed to the sample as much as possible. The silicone mold was removed and light curing is applied for another 40 seconds. The resin composite rods were polished using the same method as mentioned above. The resin cement (Multilink N, Ivoclar vivadent AG, Schaan, Liechtenstein) was mixed and injected into a hole made by one sided tape area until it reached the upper margin of the tape. Then the excess resin cement was removed using a microbrush. After that, the composite rod was placed onto resin cement with the force of 50 N using a modified durometer and the excess cement was further removed. The sample was light cured on the top surface for 40 seconds, using the protocol mentioned above. After that, the one-sided tape was carefully removed. The samples were again light cured for another 40 seconds at each side rotating at 90 degrees for each curing until it reaches 360 degrees. Next, the specimens were left for another 10 minutes to ensure complete polymerization of the samples. Finally, the samples were incubated in distilled water at 37°C for 24 hours (Incubator; DI-150, Human Lab Inc, Gyeonggi-Do, Korea) before further analysis. The finished specimen was shown on the right side of Figure 3-2.

3.7 Shear bond strength test

The adhesive area of each specimen was measured before the analysis of the shear bond strength with a digital caliper (Digital Vernier Caliper Mitutoyo CD-6 CS, Mitutoyo Co, Japan). The samples were tested for shear bond strength by a universal testing machine (AGS-X 500N, Shimadzu Corporation, Kyoto, Japan). Each specimen was fixed in the testing machine, and the shearing blade was placed parallel to the junction between the zirconia and resin cement (figure 3-3). The shear load was applied at a 0.5 mm/min crosshead speed until failure. The shear bond strength (MPa) was calculated by dividing the highest shear bond strength by the surface area of the resin cement-zirconia interface as shown in figure 3-4.

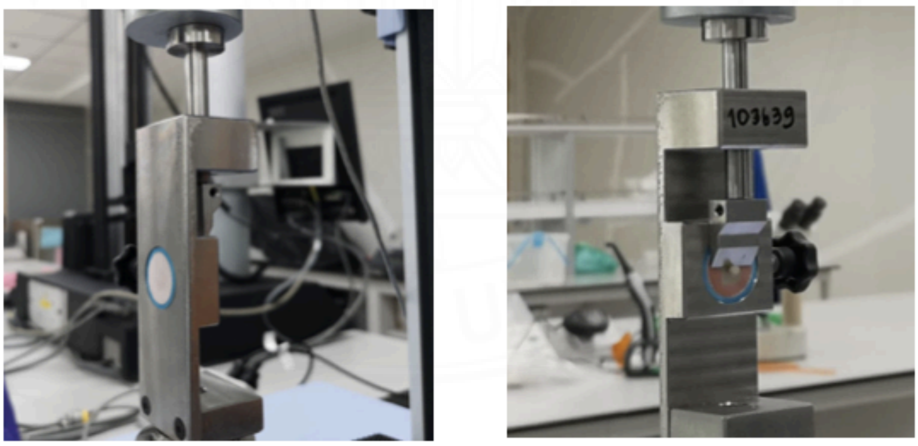

Figure 3-3 Specimen was placed in the testing jig with the universal testing machine

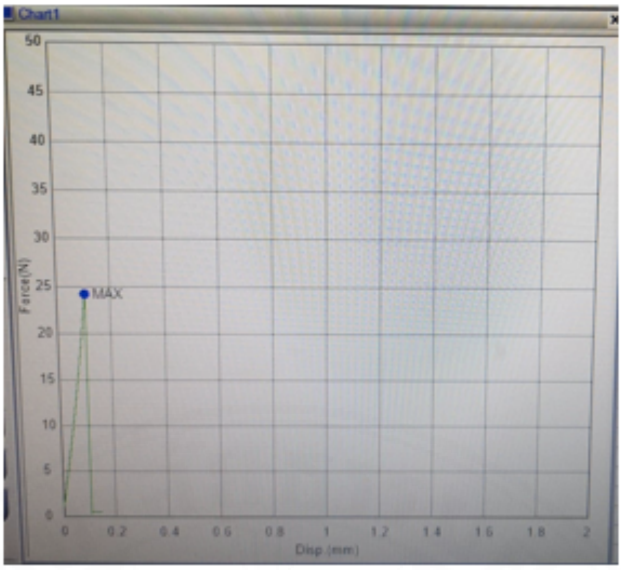

Figure 3-4 Example of maximum force (N) before dropping of graph when specimen fractured. After that the force was calculated for bond strength (Megapascal [MPa]).

3.8 Mode of failure analysis

After testing of the shear bond strength, the debonded surfaces were examined under a stereo microscope (NexiusZoom (EVO), Euromex Microscopen bv, Arnhem, Netherland) at a magnification of ×50 to evaluate the mode of failure which consist of 3 types. 1) An adhesive failure was the failure presented between zirconia and resin cement. This occurred when there was no resin cement remnant found on the zirconia surface. 2) A cohesive failure in resin cement was the failure occurred within resin cement itself. This happened when there was the whole surface of resin cement found on the zirconia surface. 3) A mixed failure was a combination of adhesive failure and cohesive failure. This presented when there was a remnant of cement attached to a zirconia surface. Furthermore, the samples from each group were randomized and examined using Scanning Electron Microscope (JSM-7800F, JEOL Ltd., Akishima, Tokyo, Japan) at magnification of x2000 to analyze the surface morphology.

3.9 Statistical analysis

The results of all groups were analyzed using SPSS 20.0 software for Mac (SPSS Inc, Chicago, Illinois, United States) setting confidence level at 95%. The normality of distribution was tested by Kolmogorov-Smirnov test (KS test) and the homogeneity of varience was tested using Levene’s test. The bond strength values were further analyzed using one-way ANOVA to assess the primary outcome and follow by Tukey’s HSD test to assess the multiple comparisons.

3.10 Expected benefits

To be able to explore the effects of cement thickness on shear bond strengths in zirconia using a universal adhesive for further clinical application.

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Result

4.1.1 Shear bond strength test

In the present study, the means and standard deviations of shear bond strength were shown in Table 4-1. Group 1 showed statistically the highest shear bond strength among all the cement thicknesses while Group 4 showed significantly the lowest shear bond strength. The Kolmogorov-Smirnov test (Table 6-1) showed that the data was normally distributed (p value ≥ 0.05) and the Levene's test (Table 6-2) was used to assess the variance (p value > 0.05).

The one-way analysis of variance (one-way ANOVA) test (Table 6-3) showed that there were statistically differences between groups (p value < 0.05) so that the hypothesis was rejected. The post-hoc multiple comparisons using Tukey HSD test (Table 6-4) demonstrated that each group was significantly different (p value < 0.05). One-way ANOVA demonstrated that the shear bond strength in the group of 50 microns cement thickness was significantly different from the cement thickness of 80, 160 and 240 ($P < 0.05$). Group 2 was significantly different from Group 1 and 3 ($P < 0.05$) while Group 3 was not significantly different ($P > 0.05$). Group 2 was significantly different from Group 1 and 4 ($P < 0.05$). Group 4 was significantly different from Group 1, 2 and 3 ($P < 0.05$) (Table 4-1).

4.1.2 Mode of failure

The distributions of failure modes were summarized in Table 4-1. Group 1 showed 90% adhesive failure with 10% mixed failure. Both Group 2 and 3 showed 70% adhesive failure and 30% mixed failure. Group 4 showed 50% adhesive failure and 50% mixed failure.

Table 4-1 Mean shear bond strengths and percentages of failure mode

Groups	**Mean shear bond strength (SD)**	**Percentage of failure mode**		
		Adhesive	Mixed	Cohesive
Group1 (50 Microns)	30.28 (3.09) [a]	90	10	0
Group2 (80 Microns)	26.86 (2.21) [b]	70	30	0
Group3(160 Microns)	25.98 (2.96) [b]	70	30	0
Group4 (240 Microns)	18.22 (1.71) [c]	50	50	0

The same letter indicates there was no statistically significant difference.

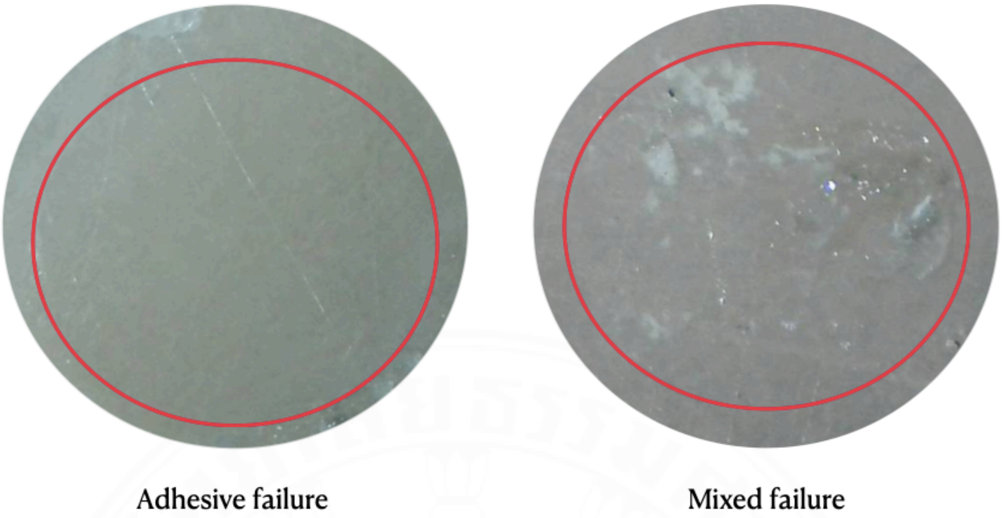

Figure 4-1 Illustration from stereomicroscope demonstrate mode of failure

The examples of macroscopic images of failure modes are presented in Figure 4-1. SEM images of specimens with mixed failure are presented in Figures 4-2. The SEM images of group 1 to group 4 were selected for analysis. The SEM images of group 4 showed a void like structure within the resin cement interface compared to group 1 to group 3. The void presented in the images may be related to the cause of early failure within the interface as the cement thickness increases. Furthermore, the chance of the resin cement presented with the void may be increased as the thickness increased.

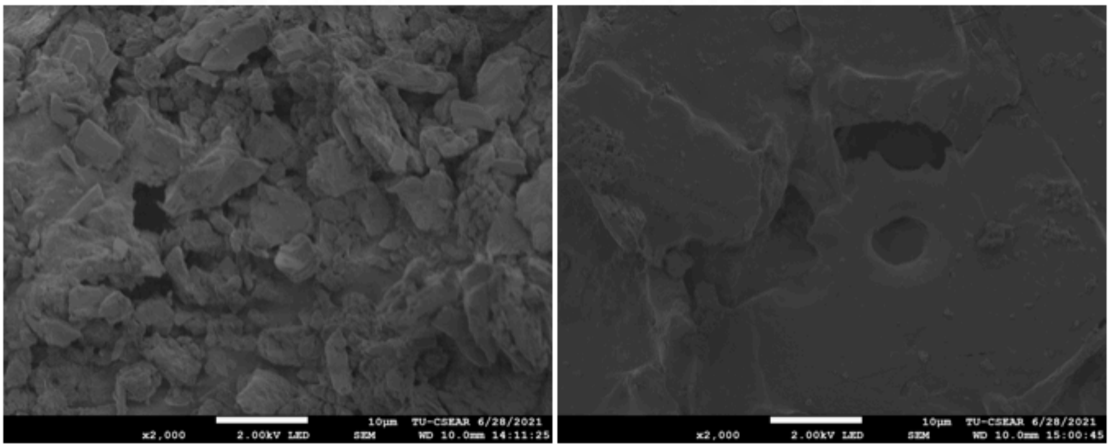

Figure 4-2 SEM image of group 4 mixed failure at high magnification x2000 within the zirconia-resin cement interface.

4.2 Discussion

The present study investigated the influence of different resin cement thicknesses on the shear bond strength of zirconia treated with a universal adhesive. The results showed that the thickness of 50 µm has the highest shear bond strength with significant differences compared to the other groups. On the other hand, the 80 µm thickness showed lower shear bond strength with significant differences with 50 µm and 240 µm while there was no significant difference with 160 µm. Group 3 (160 µm) showed significantly lower shear bond strength with group 1 (50 µm) and group 4 (240 µm) while there was no significant difference with group 3 (80 µm). Group 4 showed significantly lowest shear bond strength among all group. Therefore, the null hypothesis was rejected and the alternative hypothesis was accepted.

Many studies had examined zirconia-resin bond strength with various surface treatments [10, 11, 15]. However, the additional concern with zirconia is the internal gap or the cement space produced by the CAD-CAM manufacturing technique which might be resulted in a larger resin cement space. The marginal and internal gap of zirconia restoration can range from 60 to 240 µm according to the study from Cunali et al. in 2017 which evaluated the space for resin cement during cementation [59]. In addition, the study of Taha and Ibrahim in 2019 reported that 80 µm spacer thickness provided a significantly higher retention than 100 and 120 µm [26]. However, the study of Grajower and Lewinstein in 1983 suggested that the cement space should be set at 50 µm where it would provide the 30 µm space for the area of cement and 20 µm for a potential distortion during material fabrication [24].

So far, there is no investigation of resin cement thicknesses influencing on resin-zirconia bonding treated with a universal adhesive. The present study investigated for the first time whether the different resin cement thicknesses would affect resin-zirconia bonded with a universal adhesive. The universal adhesive incorporates various functional monomers that allow it to bond with various materials including zirconia [12]. The universal adhesive shortens treatment time and may reduce contamination risk during bonding procedure. 10-MDP, one of the functional monomer, contains both phosphate and methacrylate groups which allow these molecules to bond with both zirconia and resin [14, 19].

Therefore, our study conducted an experiment with the various cement thicknesses from 50 µm, 80 µm, 160 µm and 240 µm to prove if 80 µm, where it was mentioned in the study of Taha and Ibrahim in 2019, would still provide the highest shear bond strength when compared to the thinner resin cement thickness [26]. The 50 µm thickness represented the minimum cement space that should be set [24] whereas the 240 µm provided the highest thickness that could be found from the previous study [59]. The 240 µm thickness was also chosen from the previous studies [59] since it is the highest range of cement gap that could be reproduced by stacking 80 µm tapes.

According to the results of the present study, group 1 significantly demonstrated the highest shear bond strength. The results from our study showed a similar trend with the study from Taha and Ibrahim in 2019 where the increase in cement thicknesses resulted in a lower shear bond strength [26]. In our study, 80 µm resin cement thickness showed no significant differences in shear bond strength from the 160 µm resin cement thickness but it was significantly different from the thickness of 240 µm. The reason for the increase in shear bond strength of the thin resin cement may possibly be from the lesser proportion of voids and defects produced in resin cement during cementation process. The void that may be presented in the resin cement could start crack propagation which led to failure at lower forces. Group 4 was cemented with the thickest resin cement space and this group presented with the lowest shear bond strength. The mode of failure from the thinnest resin cement thickness (group 1) also demonstrated 90% adhesive failure and 10% mixed failure. It was observed that the more mixed failure occurred in the thicker the resin cement, the more chance of number of voids may possibly be found in the resin cement as observed in SEM (Fig 5-2). An SEM images from the study of Gradini et al in 2005, found a void within the cement layer [72]. The void presented in the resin cement could create a stress concentration when force is applied and it leads to crack initiation and eventually increases the change in failure from adhesive to mixed failure. The mode of failure from group 4 exhibited 50% adhesive failure and 50% mixed failure. Even though the group 4 demonstrated the lowest shear bond strength, the results showed high percentages of mixed failure which may not exhibit the true shear bond strength of the cement interface [29]. The mixed failure of the specimen in the present study occurred within both resin cement and the adhesive interface and it showed that the weak spot of the specimen came from either the resin cement or adhesive. To study the bond strength of the resin cement, an adhesive failure may be considered a true strength from the cement interface. In the present study, Multilink N was automated mixing from two pastes which may reduce a chance of void formation compared to other resin cement that was mixed from powder and liquid materials, ie Superbond C&B. Furthermore, a finite element experiment from Liu et al. in 2009 reported that higher stress can be developed within the resin cement when the thickness increased from 10 to 180 μm [73]. Another reason may be from the unreacted monomers within the resin cement itself that may cause the thicker resin cement to exhibit lower shear bond strength compared to thinner resin cement. A study from Kumbuloglu et al. in 2004 evaluated the degree of conversion of four resin cements ie. Panavia F, Variolink 2, RelyX Unicem Applicap, and RelyX ARC. This study reported that the degree of conversion from the dual cure resin cement range from 81% to 56% [74]. The degree of conversion from Multilink N, which is a dual cure resin cement, may have an effect on the shear bond strength of zirconia treated with the universal adhesive; however, this is yet to be studied. A sufficient amount of polymerisation of the resin cement is also an important factor to obtain the good bond strength.

From the present study, a setting of the 50 μm cement thickness may not be necessarily applied to every zirconia restoration since the bond strength of the samples were obtained only from the high translucent monolithic zirconia. The bond strength of restoration may vary when bonded with different generations of zirconia. Furthermore, this effect may only be observed when used with Multilink-N resin cement. Different resin cements have different film thicknesses and physical properties such as filler particles, viscosity and resin consistency. These properties may have an effect during cementation process which may affect the shear bond strength. Resin cement that contain phosphate monomer are able to bond with zirconia restoration. Multilink N resin cement can form a film thickness of less than 20 μm according to technical data from the manufacturer. Another recommended resin cement used with zirconia is Panavia V5 exhibited the lower film thickness of 12 μm from its technical data. From the study of Kious et al. in 2009 also founded that the thinnest film thickness of resin cement after being mixed for 3 minutes is 19 µm from RelyX Unicem [75]. The present study recommended to set up the minimum cement space of 50 µm when used Multilink N with the high translucent zirconia. However, using different resin cements with the same thickness of 50 µm may require a further study to investigate if this effect may also be applicable.

The results from the present study may be applied in the clinical situation when setting up a cement space during CAD design of zirconia restoration, where the thinner cement thickness may provide a better bond strength. However, it may rise a fairy high expectation where the tooth abutment must be precisely prepared in order to obtain a desirable cement space. Thinner cement space can lead to a lesser room of error when seating the restoration. Inadequate tooth preparations could create undercuts resulting in thicker cement thicknesses to correct the path of insertion. This could eventually lead to lower bond strength. Thus far, the present study demonstrated the first time that different resin cement thicknesses affected shear bond strengths of zirconia treated with a universal adhesive and that a 50 µm resin cement thickness provided the highest initial bond strength after incubated for 24 hours. However, the long term of shear bond strength in this setting is not yet studied.

Within the limitation of the present study, it can be concluded that the thin space of 50 µm of resin cement thickness exhibited the highest shear bond strength while the thicker space of 240 µm demonstrated the lowest shear bond strength when zirconia was treated with a universal adhesive. Further studies should be investigated on different resin cement thicknesses of zirconia treated with different bonding agents.

CHAPTER 5
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

Within the limitation of the current study, the following conclusions can be drawn:

50 µm of resin cement thickness exhibited the highest shear bond strength while the 240 µm demonstrated the lowest shear bond strength when a high translucent zirconia was treated with a universal adhesive.

5.2 Limitations

1) The current study was vitro study, that may be not be directly applicable in the clinical situation.

2) For the shear bond strength test in the present study, the thick shear blade is only available. Using a thinner shear blade may vary the shear bond strength values.

5.3 Future studies

1) Shear bond strength of zirconia bonded with dental cement using different types of bonding systems.

2) Shear bond strength of zirconia bonded with different type dental cements with different thicknesses.

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APPENDICES

APPENDIX A

Shear bond strength test

Normality test

The Kolmogorov-Smirnov test (Table 6-1) showed that there was accept the hypothesis that the data was normally distribution (p value ≥ 0.05).

Table 6-1 The Kolmogorov-Smirnov

group		**Kolmogorov-Smirnov**[a]			**Shapiro-Wilk**		
		Statistic	**df**	**Sig.**	**Statistic**	**df**	**Sig.**
Thickness	50	.148	10	.200*	.914	10	.308
	80	.181	10	.200*	.941	10	.559
	160	.197	10	.200*	.920	10	.361
	240	.156	10	.200*	.980	10	.966

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Homogeneity of variance test

The Levene's test (Table 6-2) showed that there was accept the hypothesis that the data was equal in variance (p value > 0.05).

Table 6-2 Levene's Test

	Levene Statistic	**df1**	**df2**	**Sig.**
Thickness	3.664	3	36	.058

Analysis of variance

The One way analysis of variance (One way ANOVA) test (Table 6-3) showed that there was reject the hypothesis that the data was significantly different of each group (p value < 0.05).

Table 6-3 The One way analysis of variance (One way ANOVA) test

	Sum of Squares	**df**	**Mean Square**	**F**	**Sig.**
Between Groups	778.014	3	259.338	39.699	.000
Within Groups	235.175	36	6.5333		
Total	1013.190	39			

Multiple comparisons

The post-hoc multiple comparisons applying Tukey HSD test (Table 6-4) was assessed which groups were significantly different (p value < 0.05).

Table 6-4 The post-hoc multiple comparisons applying Tukey HSD test for shear bond strength

Dependent Variable: Thickness
Tukey HSD test

(I) Formulation		Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
50	80	3.425*	1.14304	.024	.3465	6.5035
	160	4.301*	1.14304	.003	1.2225	7.3795
	240	12.060*	1.14304	.000	8.9815	15.1385
80	50	-3.425*	1.14304	.024	-6.5035	-.3465
	160	.876	1.14304	.869	-2.2025	3.9545
	240	8.635*	1.14304	.000	5.5565	11.7135
160	50	-4.301*	1.14304	.003	-7.3795	-1.2225
	80	-.876	1.14304	.869	-3.9545	2.2025
	240	7.759*	1.14304	.000	4.6805	10.8375
240	50	-12.060*	1.14304	.000	-15.1385	-8.9815
	80	-8.635*	1.14304	.000	-11.7135	-5.5565
	160	-7.759*	1.14304	.000	-10.8375	-4.6805

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