BOND POTENTIAL OF LITHIUM DISILICATE TO HEAT-CURED POLYMETHYLMETHACRYLATE (PMMA)

by

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CERTIFICATE OF APPROVAL

MASTER'S THESIS

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ABSTRACT

BONDING OF LITHIUM DISILICATE TO HEAT-CURED POLYMETHYLMETHACRYLATE (PMMA) JAMES GWAU-CHIAHNG LINKOUS DMD, PROSTHODTONICS, 2015

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Introduction: Implant-supported, implant-retained hybrid prostheses is an increasingly common restoration for edentulous patients. Reviews of the literature have shown that mechanical failure mode between the denture tooth and the denture base is a common problem. To decrease the possibility of failure, lithium disilicate has been suggested as a restorative material for the hybrid prosthetic tooth. This new design has created an untested interface between lithium disilicate and polymethylmethacrylate (PMMA). Objective: This study will compare the bond potential between lithium disilicate and heat-cured PMMA with various surface treatments. Method: 30 specimens, 3 test groups (N=10) with different surface treatments on lithium disilicate were created: no treatment (Control) hydrofluoric acid etch (HF), hydrofluoric acid etch and ceramic priming agent (HF+Primer). Specimens were connected to heat-cured PMMA with constant 30 psi pressure and allowed to cure to manufacturer's instructions. Specimens were shear loaded on a MTS Insight at a rate of 0.01mm/s. Results: Test group Control had a mean shear load of 7.1 ± 2.8 . Test Groups HF and HF + Primer had a much greater shear load with mean values of 21.1 ± 3 and 22.1 ± 2.9 , respectively. One-way ANOVA statistical

test was used obtaining a p-value < 0.001, demonstrating a difference between the test groups. A Bonferroni post hoc test was preformed resulting in a p-value < 0.001 when comparing the Control with test group HF and the Control with test group HF + Primer. No significant difference was found between test groups HF and HF + Primer. **Conclusions:** Evidence suggests that acid etching the ceramic is the most significant factor in achieving a bond between lithium disilicate and heat-cured PMMA. The additional step of a priming agent may be unnecessary since it did not increase the shear bond strength in our study.

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CHAPTER I: INTRODUCTION

The critical evaluation of denture prostheses is important because, although the percentage of the denture wearing population has declined, due to population growth, the overall number of denture wearers will increase (National Center for Health Statistics, 1994). This assessment means that denture prostheses will continue to be in demand in the future. The number of U.S. adults needing complete dentures is expected to increase from 35.4 million in 2000 to 37.9 million in 2020 (Douglass, Shih, & Ostry, 2002). Further study on the efficacy, strength, and longevity of denture prostheses, as well as patient satisfaction, must be done as a scientific endeavor in order to the meet the needs of this demographic (Hummel, Wilson, Marker & Nunn, 2002). Properly fitting dentures have been shown to be an indicator of the quality of life for a patient (Critchlow & Ellis, 2010), and will continue to be a treatment option for many patients.

A common problem with denture prostheses is tooth debonding from the denture base. The failure rate of acrylic resin dentures resulting from fracture has been reported to be unacceptably high (Cunningham, 1993; Vallittu, Lassila & Lappalainen, 1993), with the most common type of failure encountered being debonding or fracture of the teeth (Darbar, Huggett & Harrison, 1994). Some sources have suggested that approximately 30% of all denture repairs received by commercial dental laboratories involved faults attributed to failed bonding between the teeth and the denture base resin (Huggett, John, Jagger & Bates 1982; Vallittu, Lassila, & Lappalainen, 1993). It was hypothesized that as implant use becomes more prevalent, denture tooth debonding or fracture may be an even greater clinical problem. The lack of proprioceptive ability with implant prostheses would allow for increased bite force and thus exacerbate denture tooth failure (Gunne, Rangert, Glanz & Svensson, 1997; Lindquist & Carlsson, 1985). Later literature supported this presumption and showed that the prevalence of tooth debonding or denture veneer fracturing is one of the most common complications with implant prostheses (Goodacre & colleagues, 2003; Walter & MacEnter, 1994; Davis, Packer, & Watson, 2003). The literature clearly shows that this problematic interface is in great need of advancement in order to reduce the number of denture prosthesis failure.

CHAPTER II: REVIEW OF THE LITERATURE

DENTURE BASE TYPES AND CHEMISTRY

According to the International Organization of Standards (EN ISO) Specification 20795-1 (2008), denture bases can be classified into five types:

Type 1- heat-curing polymers

Type 2 - self-curing polymers

Type 3- thermoplastic materials

Type 4 - light-curing materials

Type 5 - microwaved polymerization

Except for Type 4 polymers, which are composed of urethane dimethacrylate, each of these systems has PMMA as the principal polymer. Type 1 polymers are the most prevalent denture base material and denture tooth material. It shows great strength, dimensional stability, and durability (Cunningham, 2000; Zarb & Bolender, 2004). PMMA is a long carbon chain matrix that can contain multiple other components in the matrix to help improve its physical and chemical properties. For example, certain PMMA matrices have added butadiene-styrene which is a synthetic rubber to increase the strength of the material (Rodford R, 1986).

For self-curing PMMA, the initiator is commonly benzoyl peroxide or disobutylnitrole and the activator is a tertiary amine. Heat-cured

polymethylmethacrylate has similar initiators as the self-cured polymers, but the energy source, the activator, is heat as oppose to the tertiary amine. Light-activated denture base polymers are urethane dimethacrylate (UDMA) with a photoinitiator system. The basic steps for the polymerization of these molecules reaction are as follows: initiation, propagation, and termination. Initiation uses the catalyst to form a free radical of the matrix monomer. Propagation creates a chain of monomers, continuing the polymer formation until steric hindrance or termination of the polymer formation (Zarb & Bolender, 2004).

Plasticizers are used to alter the properties of the resin polymer. Examples of plasticizers include dibutyl phthalate, silica (SiO2) fibers, and polycrystalline structures such as zirconium silicate. These plasticizers can decrease the thermal coefficient of expansion, increase stiffness, and increase thermal diffusivity. Opacifiers and dyes are used in order to change the optical properties of the resin material (Zarb & Bolender, 2004).

Microwave-cured denture base materials show promise as an alternative to the heat-cured method. They have shown satisfactory strength and durability (Al-Hanbali, Kelleway, & Howlett, 1991; Sanders, Levin, & Reitz, 1991; Vallittu & Ruyter, 1997). The main advantage of microwave-cured denture base materials is that the process decreases the polymerization time, making them at more desirable for the clinician (Polyzois, Karkazis, Zissis, & Demetriou, 1987).

Light-polymerized urethane dimethylmethacrylate (UDMA) has shown similar mechanical properties to the traditional heat-cured PMMA denture base (Ali, Yunus, &

Abu-Hassan, 2008; Diaz-Arnold, Vargas, Shaull, Laffoon, Qian, 2008; Machado & colleagues, 2007). This resin system eliminates the need for flasking, boil-out, and long processing times. Instead, the base is light-cured while the teeth are placed with a VLC set-up resin. Then contour resin is used for festooning, creating the gingival contours of the denture. All phases of the denture are composed of UDMA and cured by visible light.

DENTURE TEETH

Until around 1960s complete denture teeth were porcelain. Acrylics had not yet been developed and porcelain was the only material known to meet the esthetic demands of patients in the early 1900s. This type of feldspathic porcelain was very weak and brittle (Hodson, 1959). As a result, porcelain denture teeth were susceptible to fracture. Moreover, the abrasive nature of this type of ceramic made it harmful to opposing natural dentition (Boddicker, 1947; Koran, 1972). Due to feldspathic porcelains low resiliency and high modulus of elasticity, patients often complained of "clicking" noises when they talked or enacted parafunctional habits (Zarb & Bolender, 2004).

PMMA denture teeth were later introduced and these denture teeth provided a few advantages over porcelain denture teeth. Unlike porcelain teeth, PMMA denture teeth bonded to the denture base (Schoonover, 1952; Spratley, 1987, Suzuki, Sakoh & Shiba, 1990). In addition, they had a natural feel because of the lack of "clicking" noise that was evident with porcelain teeth. They were considered tough, but easy to grind and polish after occlusal adjustments were made. Also, they were non-abrasive; instead of abrading the opposing dentition, PMMA denture teeth were more likely to wear away (Koran, Craig, & Tillitson 1972; Harrison & Huggett 1975; Eckfeldt & Oilo, 1989). The first PMMA teeth did not have a cross-linking agent between the PMMA polymer chains. The introduction of cross-linking, or covalent bonding between PMMA polymer chains, added an additional component to the denture teeth that reduced, if not eliminated, blanching and crazing (Korkmaz, Dogan, Dogan, & Demir, 2011). As a result, PMMA teeth are widely used as the denture tooth of choice.

DENTURE BASE POLYMERIZATION AFFECTING BONDING

The method of denture base resin polymerization might also influence the adhesion between acrylic teeth and the acrylic resin denture base (Cunningham & Benington, 1999). Polymerization by microwave energy is an alternative to conventional water-bath processing (Schneider, 1995; De Clerk, 1987) presenting the principal advantage of greatly reducing the polymerization time of the denture base resin (Schneider, Curtis, & Clancy, 2002). Heat from the exothermic polymerization and the source to cure the material can be above the boiling point of the monomer, leading to the formation of porosities (De Clerk, 1987), which can also reduce the strength properties of the denture base material (Gettleman Nathanson & Myerson 1977; Keller & Lachtenschlager, 1985). Polyzois and Zissis (1995) found inferior bonding between acrylic teeth and denture base material polymerized by microwave energy, as compared to hot water-bath polymerization. The formation of porosities at the interface between the artificial teeth and denture base resin after microwave polymerization was a probable

explanation for the bond failure (Polyzois & Dahal, 1993). Schneider, Curtis and Clancy (2002), as well as Takahashi and colleagues (2000), reported similar results. Higher bond strengths between the denture teeth and denture bases were obtained with conventional heat-polymerized acrylic resin than with microwave-polymerized acrylic resin. However, in another study, the bond strength of microwave-polymerized acrylic resins to denture teeth was greater than that of conventional heat-polymerized acrylic resins (Geerts & Jooste, 1993). Differences in the idiosyncratic properties of each type of denture teeth, acrylic resins, and the experimental techniques used might explain the variation in reported results (Barpal & colleagues, 1998).

Visible-light polymerized UDMA denture bases have exhibited decreases in denture base to denture tooth bond strengths (Clancy, Hawkins, Keller & Boyer, 1991; Kawara, Carter, Ogle & Johnson, 1991; Cunningham, 2000). It is unsure as to why there is lower denture tooth to denture base bonding for UDMA, but authors have hypothesized that the addition of organic solvent or monomer does not wet the denture base in the same manner for light-polymerized denture bases as it does for heat-cure denture bases (Clancy, Hawkins, Keller & Boyer, 1991; Kawara, Carter, Ogle & Johnson, 1991; Cunningham, 2000).

Deficient laboratory procedures may also prevent adequate bonding between the tooth and denture base resin, causing subsequent bond failures. (Cunningham & Benington 1997; Huggett & colleagues, 1982). Wax residue contamination on the tooth ridge-lap surface may cause significantly weaker bonds between teeth and denture base resin (Cunningham & Benington, 1999; Spratley, 1987). Careless application of the

separating medium during processing (Rupp, Bowen & Paffenberger, 1971) and insufficient available monomer can affect the denture tooth bond as well.

PMMA DENTURE TEETH AND DENTURE BASE INTERFACE

MECHANICAL RETENTION

There have been many attempts to increase the retention of denture teeth with mechanical means. Fletcher, Al-Mulla, Amin, Dodd, and Ritchie GM (1985) reported increased shear and tensile bond strengths between PMMA denture teeth and denture bases simply by roughening the interfacial surfaces of the denture base and the denture tooth. Chung, Chung and Chan (2009) examined the effects of grinding and sandblasting on denture tooth bonding and showed a very effective means of increasing bond strength (Chung, Chung, & Chan, 2009). Cardash, Liberman, and Helft (1986) and Cardash, Applebaum, Baharav, and Liberman (1990) created vertical retention grooves, also known as diatorics, into the denture teeth and found an increase in retention of denture teeth to the PMMA denture base.

The literature demonstrates that there is an increase in bond strength through mechanical retention by methods such as ridge-lap surface modification from bur abrasion or bur grooving, diatorics, aluminum oxide particle abrasion, and/or tribochemical coating (Cardash & colleagues, 1990; Cunningham & Benington, 1999; Spratley, 1987; Chung, Chung, & Chan, 2009; Nishigawa & colleagues, 2006; Consani, Carmignani, Mesquita, Correr-Sobrinho, & Guiraldo, 2010; Consani, Naoe, Mesquita, Sinhoreti, & Mendes, 2011; Saavedra & colleagues, Vallittu, 1995; Vallitu, Ruyter & Nat, 1997; Vallittu & Ruyter, 1997; Vallittu, 2009).

CHEMICAL RETENTION

Many studies have attempted to increase the bonding between the denture tooth and denture base by chemical means. The results have shown some promise. Takahashi, Chai, Takahashi, and Habu (2000) studied both mechanical and chemical means of increasing denture tooth bond and did not find a significant increase in retention of denture teeth with the addition of diatorics, but greater retention with a solvent, specifically dichloromethane. Applying methyl methacrylate monomer liquid to the denture base or denture tooth before denture processing has resulted in increased retention or failure loads ((Huggett, John, Jagger & Bates, 1982; Barpal, Curtis, Finzen, Perry & Gansky, 1998; Rached & Del Bel Cury, 2001; Geerts & Jooste, 1993; Papazoglou & Vasilas, 1999, Barbosa & colleagues, 2009). The theory proposed for this increase in retention is that the methyl methacrylate monomer liquid and allows for better wetting and/or dissolves the surface layer, allowing for a better diffusion and bond between the denture base and the acrylic denture tooth. Similar results have been reported with other organic solvents such as chloroform (Shen, Colaizzi, & Birns, 1984), acetone (Rached & Del Bel Curry, 2001; Rached, Powers, & Del Bel Cury, 2004), and methylene chloride (Minami, Suzuki, Minesaki, Kurashige & Tanaka, 2004; Sarac, Sarac, Kulunk & Kulunk, 2005). This chemical means for bonding is available for both lightcured UDMA and heat-cured PMMA denture bases (Cunningham, 2000; Hayakawa, Hirano, Nagao, Matsumoto & Masuhara, 1991; Yanikoglu, Duymus & Bayindir, 2002).

SILICA-BASED CERAMICS AND DENTURE BASE INTERFACE

Silica-based ceramics can be categorized by composition: feldspathic (SiO2-Al2O3-Na2O-K2O), leucite-reinforced (SiO2-Al2O3-K2O3), lithium-disilicate (SiO2-LiO2) (Conrad Seong & Pesun, 2007; Kelly 2008). These glass-ceramics are etched with 5% to 9.5% hydrofluoric acid for 1 minute and then coated with a silane coupling agent. The etchant increases the surface free energy and surface area, enhancing the bond capacity of the substrate (Chen, Matsumura & Atsuta, 1998; Ahmad, 2002). Before cementation, a silica-based ceramic is extremely brittle, showing weak tensile and comparatively low flexural strength; however, once these restorations are bonded with a resin composite cement, their strength greatly increases (Blatz, Sadan, & Kern 2003; Kelly 2008). This demonstrates the potential of bonding ceramics not only helps with retention but increases strength, making adhesion of ceramics an important procedure to implement for the longevity of a prosthesis.

Silane coupling agents have been used in many instances to create adhesion between an inorganic and organic substrate. The most common silane coupling agent applied in dental laboratories is 3-trimethoxysilylproprylmethacrylate (MPS). It's mechanism of adhesion by condensation, good wetting, and chemical bonding between it and the inorganic substrate is well theorized in the literature (Matinlinna & colleagues, 2004; Clark & Plueddemann, 1963; Ishida, 1984; Umemoto & Kurata, 1995). The silane molecule aggregates near the inorganic substrate and appropriately arranges near each monomer through hydrogen bonding. By what's called silane condensation, the silane molecule forms siloxane bonds, -Si-O-Si-, between each other creating the larger polymer chain. The siloxane molecule's hydroxyl group reacts with the inorganic substrates hydroxyl group forming a –Si-O-Metal- chemical bond, releasing water as a product of the chemical bond formation. The organic functional group of the silane molecule is the vinyl group, creating a bond between the silane molecule and the resin matrix (Matinlinna & colleagues, 2004).

Silica-based ceramics have in the past been examined as a dental restorative treatment for denture teeth. Because of the poor fracture toughness and flexural strength of the older porcelain denture teeth, bonding studies that examined the bond of porcelain to PMMA mainly showed cohesive fractures within the porcelain (Paffenbarger, Sweeney, & Bowen, 1967). Also, there seemed to be an inverse relationship between bonding capabitilies and ceramic strength. The higher the bond to the denture base had a decrease in the strength of the feldspathic denture teeth. (Semmelman & Kulp, 1968). It was soon later theorized that even though the bond potential between PMMA and feldspathic porcelain was strong, the polymerization shrinkage and shear stress applied during cooling of the heat curing PMMA produced unfavorable stresses on the porcelain leading to the cohesive failures of the ceramic (Myerson, 1969). In another attempt to measure the bonding capabilities of silica-based ceramics to PMMA, Takahito and colleagues (2000) added MPS into the mixture of PMMA denture resin in a ratio of 94/6 (mol%), PMMA to MPS respectively. They too showed fractures within ceramic and great bond potential, above 20 MPa, between the two substrates when using MPS as a silane coupling agent. With the increased flexural strength (~320-440 MPa), hardness (~5.5GPa), and fracture toughness (~2.5-3 MPa) of lithium disilicate ceramics (Albakry,

Guazzato, Swain, 2003; Deng, Miranda, Pajares, Guiberteau, & Lawn, 2003; Lawn & colleagues, 2004), the weak component of previous studies causing failure can be challenged. The stronger ceramic may be durable enough to more accurately measure the bond strength between PMMA denture resin and silane coupled silica-based ceramics rather than cohesively fracture.

SUMMARY

If the problem of debonding denture teeth were solved, the edentulous population would benefit by avoiding repeated repair of their denture prostheses. This benefit would decrease chair time, which would decrease the cost to the dentist, and to the patient as well.

There have been many attempts to increase this bond strength, attempting to diminish the prevalence of denture prosthesis failure. Mechanical and chemical methods have both been examined to increase the bond strength of the denture tooth to denture base. Multiple methods such as diatorics, sandblasting, monomer wetting, bur grooving have all been methods to increase the bond between denture teeth and denture base. Silica-based ceramics have been used in the past as a denture tooth restorative treatment, but due to the ceramics poor strength and the high incidence of cohesive failures, the overall prognosis was unfavorable.

This study varies from the literature because lithium disilicate ceramics are substantially different from the at-the-time feldspathic ceramics used in studies to measure porcelain to acrylic bonding. Lithium disilicate ceramics have higher values in flexural strength, toughness, and hardness, decreasing the likelihood of a cohesive fracture. These physical properties of lithium disilicate ceramics make the ceramic a possible candidate as a denture tooth restorative treatment; however, the bonding capability between these two substrates has not yet been tested. The purpose of this study is to measure the bond strength of lithium disilicate to heat-cured PMMA with various surface treatment applications.

CHAPTER III: MATERIALS AND METHODS

For this in vitro investigation, 30 specimens were fabricated. Three experimental groups with different surface treatments were devised with 10 specimens in each group:

1) C (Control): Lithium disilicate cylinders with no surface treatment.

2) HF: Lithium disilicate etched with 4.8% HF acid.

3) HF + Primer: Lithium disilicate etched with 4.8% HF acid and a coating of trimethoxysilypropyl methacrylate (MPS).

12.50 mm diameter cylindrical lithium disilicate (E.MAX, Ivoclar USA) specimens were treated appropriately to their test groups. Test group C received no treatment. Test group HF received 4.8% HF acid etch for 20 seconds followed by 60 seconds distilled water rinse. They were then placed in an ethanol solution and in an ultrasonic for 5 minutes. Test group HF + Primer received the same acid etching protocol followed by application of MPS (Monobond S, Ivoclar USA) and 60 seconds to air dry.

A 3-piece titanium holder was 3-D printed using the ARCAM A1 which uses electron beam melting technology. The ceramic was treated and placed into the base of the holder and a polyvinylsiloxane (PVS) spacer will be placed around the ceramic in order to aid in the separation of the specimen from the holder base. PMMA (Lucitone 199, Dentsply USA) was mixed to manufacturer's instructions and injected into the hollowed square after the hollow square is placed on the base holder.



Figure 1: The 3-D Printed Titanium holder, hollow square, and cap.





Figure 2: Treated ceramic placed into the holder and PVS injected around as a spacer. Excess PVS was removed.

Figure 3: PMMA being injected into the titanium hollow square. Filled until there was excess material. The cover will be placed on top and compressed to 30 PSI and held together with a Cclamp. Excess PMMA will be removed and PVS will be injected around the microgaps in order to prevent moisture contamination from the water bath. The unit will be heat cured under slow-cure protocol which consists of an 8 hour cure and a gradual increase in temperature to 212 degrees Farenheit.



Figure 4 (Left): The final titanium printed unit with the excess PMMA removed. Will be placed into the water bath and heat cured to manufacturer's instructions.

Figure 5 (Below): A complete set of one of the test groups.



Shear bond strength will be tested In order to ensure specimens are loaded at the same location, a custom made titanium specimen holder will be 3-D printed using the ARCAM A1. The specimen will be placed and clamped together by hand tightening a screw and a hexagonal nut fastener. Shear bond strength will be tested on an MTS Insight machine with a knife edge blade at a data acquisition rate of 60 hz, a preload of 1N, and a load speed of 0.01mm/s.

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Figure 6: Specimen holder clamping down on a specimen using a screw and fastener and placed in the MTS Insight.

CHAPTER IV: RESULTS

Groups HF and HF + Primer had much higher failure modes than the Control. (Table 1) The mean shear failure load of the Control was 7.1 ± 2.8 MPa. The mean shear failure load of test group HF was 21.1 ± 3 MPa. The mean shear failure load of test group HF \pm Primer was 22.1 ± 2.9 MPa. (Figure 1) An unpaired One-way Anova test was performed, resulting in a p-value less than .001 which leads us to reject the null hypothesis and suggesting that there is a difference between the test groups. A post hoc Bonferroni test was also performed, individually comparing the test groups. A p-value of less than .001 was obtained when comparing the Control to test group HF and when the Control was compared to HF + Primer. There was no significant difference between the test groups HF and HF + Primer with a p-value of 0.42. Specimens were examined under increased magnification with a hirox digital camera to assess failure mode. Failure modes appeared to be adhesive for all test groups. There were increased porosities on the surface of group HF + Primer.

Table 1.



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Figure 8:	
Control	
specimen.	



Figure 9: Control specimen. At increased magnification

Figure 10: HF Specimen



Figure 11: HF Specimen at increased magnification



Figure 12: HF + Primer Under Magnification



Figure 13: HF + Primer Specimen at increased magnification



Figure 14: Comparison of specimen from test group HF(Right) and HF + Primer (Left)

CHAPTER V: DISCUSSION

Our evidence suggests that there is a bond potential between lithium disilicate and PMMA when the surface is appropriately treated. We did not find a statistical difference between the test groups HF and HF + Primer. These findings lead us to conclude that the most important factor in achieving a bond is acid etching the ceramic. Typically with ceramic bonding, a silane coupling agent increases the bond strength when bonding to a ceramic (Matinlinna & colleagues, 2004), so it was unexpected to have test groups HF and HF + Primer to have similar bond strengths. A couple of explanations have been hypothesized. One hypothesis is that test groups HF and HF + Primer had different failure modes. While they both may have had adhesive failure modes, test group HF may have a failure mode between the ceramic and the PMMA while test group HF + Primer may have a failure mode between the silane and the PMMA. In order to determine this, additional analysis with scanning electron microscopy would be necessary for it was difficult to determine the difference between surfaces when multiple materials had coated that surface, like for the test group HF + Primer. The second hypothesis is that the acetone which is the solvent for the methylmethacrylate may have dissolved away the primer on the ceramic, thereby reducing the presence and thus effects of the silane coupling agent. This hypothesis might help explain why under magnification we see an increase in porosity like shapes on the surface of specimens from HF + Primer. The third hypothesis is that you achieve adequate wetting of the ceramic with or without a primer, implying less of a chemical bond and more of a micromechanical bond.

CHAPTER VI: CONCLUSION

The purpose of this study was to examine the bond potential between lithium disilicate and heat-cured PMMA. Our evidence suggests that acid etching the ceramic is the strongest contributor to achieving a bond. A silane coupling agent may be used but is not necessary. This data is in concordance with previous studies examining this interface is well as preliminarily supports the clinical use of an alternative hybrid design with lithium disilicate teeth.

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