Effect of post-contamination surface treatment on the bond strength of adhesively bonded ceramic indirect restorations

by

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ABSTRACT

Effect of post-contamination surface treatment on the bond strength of adhesively bonded ceramic indirect restorations

Drew Krena, DMD, 2017

Thesis directed by: Douglas Dickinson, PhD, Research Advisor, Dental Materials

Purpose: The purpose of this research was to determine the effects of contamination, different cleaning methods, specifically Ivoclean, and simulated aging on the shear bond strength (SBS) of resin cement to 2 different ceramic materials, zirconia and lithium disilicate.

Materials and Methods: Blocks of lithium disilicate and zirconia were prepared for bonding by hydrofluoric acid and particle abrasion respectively. The samples were divided into 3 groups for each type of ceramic:, Ivoclean pilot study, 24 hour storage group, and the thermocycled group. The Ivoclean pilot study consisted of 10 uncontaminated samples and 10 uncontaminated Ivoclean treated samples for both ceramics. The 24 hour stored and thermocycled groups were broken down to 5 subgroups: saliva uncontaminated, contaminated, Ivoclean, phosphoric acid (lithium disilicate) or air abrasion (zirconia), and air/water spray, with each subgroup containing 10 samples. The samples were tested on an universal testing machine to determine the SBS.

Results: The SBS of uncontaminated samples treated with Ivoclean was not statistically different from the uncontaminated control samples. The 24 hour stored samples were not significantly different from each other, except for the contaminated samples, which were significantly lower than uncontaminated. The thermocycled groups showed somewhat more difference. A significant number of samples debonded. Interestingly, all of the Ivoclean treated zirconia samples debonded and SBS could not be recorded. Lithium disilicate uncontaminated controls and the phosphoric acid cleaned samples were statistically similar. The thermocycled uncontaminated control and air abraded samples proved to be statistically similar.

Conclusions: Saliva contamination and simulated aging (thermocycling) decreased bond strengths independently from each other and compounded one another. Cleaning methods generally did not return bond strengths to that of the uncontaminated samples. Evidence was not found to support the use of Ivoclean for either zirconia or lithium disilicate, although there was a trend to a modest benefit with zirconia. Air abrasion for zirconia and phosphoric acid for lithium disilicate maintained bond strengths not significantly different from the uncontaminated samples following thermocycling.

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CHAPTER 1: Introduction

STATEMENT OF THE PROBLEM

Ceramic indirect restorations are increasingly becoming the choice for crowns due to esthetic considerations and structural properties of the materials. However, regardless of the material used, the restoration must be cemented in place, and the bond strength of a permanent indirect restoration is critical to the success of the restoration. The bond of the cement to the ceramic crown imparts strength to the restoration by preventing micro fractures from propagating from the intaglio surface. Bond strength only gains its maximum potential through a precise, controlled bonding technique. Imprecise technique or contamination can impede the bond, potentially decreasing the lifespan of a restoration. Cleaning methods have been proposed to remove contamination and restore bond strength. A clear understanding of the material to be bonded, the type of cement used, and the technique to bond the materials are paramount.

Ceramic restoration materials can be divided into two broad subgroups: silica-based glass ceramics and non-silica-based ceramics[1]. The latter include zirconia and alumina, while the former consist of lithium-disilicate, feldspathic, lithium monosilicate and leucite-reinforced. With the advent of novel materials, new bonding protocols have been developed. The bonding protocol for silica-based glass ceramics and non-silica-based ceramics is different due to the materials composition. Silica-based glass ceramics use hydrofluoric acid to etch the bonding surface due to its effect on the silica particles. Non-silica-based ceramics, lacking these particles, will not etch if acid is applied. Therefore, a different bonding approach must be utilized.

Contamination during the bonding process can degrade the final bond strength. A common source of contamination is with saliva during try-in, which can affect the final bond adversely due to salivary proteins on the intaglio surface of the crown that inhibit cement binding sites. Therefore, the restorations must be cleaned appropriately before cementing in place, but in a manner that itself does not reduce bond strength by chemical modification of the surface. Thus, the cleaning technique must be geared to the chemistry of the crown material and the bonding agent.

There is also a need to simplify the cementation/bonding process. This standardization becomes important for dentists due to the economics of materials, patient treatment time, and the technique sensitivity of the cementation/bonding process. One area of the bonding protocol that can be simplified is the cleaning of the prosthesis after try-in.

Ceramic cleaning methods after try-in procedures have a significant influence on the resin-ceramic bond strength. Certain cleaning methods have been advocated to remove intaglio surface contaminants. Etching with phosphoric acid has been put forward as an effective cleaning method for silica-based glass ceramics; however, cleaning with phosphoric acid has shown to inhibit/reduce bonding in zirconia due to an overconcentration of phosphate molecules that inhibit binding sites of the bonding agent used. A supersaturated zirconia particle, sodium hydroxide based solution, Ivoclean, was introduced with the goal of creating an ideal intaglio surface for the bonding procedure.

With the introduction of a new cleaning mechanism, the bond strength must be examined to ensure the successful cleaning of the intaglio surface and maintenance of overall bond strength. Treatment with Ivoclean has been advocated as a universal cleaner to remove contaminants following try-in. However, this cleaning agent has not been evaluated fully for zirconia-based restorations and more generally as a replacement for current phosphoric acid cleaning in other silica-based glass ceramic materials.

SIGNIFICANCE

If Ivoclean is used to treat the restoration prior to bonding to clean the debris, then bond strengths should be consistent with a clean, uncontaminated surface. Ivoclean may be a suitable cleaning agent for all ceramic restorations that will provide a single cleaning approach to be standardized throughout the Army Dental Corps, thereby reducing the cost of materials for cleaning indirect restorations after try-in and increasing the success and longevity of the indirect ceramic restoration by optimizing the cementation/bonding.

Chapter 2: Review of the Literature

DENTAL CERAMICS

Dental ceramics have become increasingly the choice for indirect restorations due to their esthetic and structural properties. Ceramic restoration materials can be divided into two different subgroups: silica-based glass ceramics and non-silica-based ceramics[1]. Kelly[2] described dental ceramics in three different categories based on their composition; predominantly glass, particle-filled glass, and polycrystalline. Predominantly glass and particle-filled glass ceramics can generally be grouped under silica-based ceramics, while polycrystalline ceramics describe the non-silica-based ceramics. Each group has different attributes and properties that lend themselves to different clinical situations that will be further discussed. Kelly[2] also finds it important to understand that any dental ceramic within these categories is also considered a composite, meaning a composition of two or more entities. The addition of materials into the glass matrix or the crystalline structure will impart different properties to the ceramics.

Silica-based ceramics

The silica-based ceramics can be classified as predominantly glass and particle-filled glass. Predominantly glass dental ceramics best mimic the optical properties of enamel; an example is feldspathic porcelain. Particle-filled glass dental ceramics add filler particles to the base glass composition that improve mechanical properties; examples include leucite-reinforced and lithium disilicate glass ceramics[2]. These ceramics are characterized by brittleness and limited flexural strength, but have a propensity to increase fracture resistance after being adhesively luted to the tooth[3]. Lithium disilicate glass ceramics have improved physical properties compared to other glass ceramics. Lithium disilicate glass ceramic has a flexural strength of 360-500 MPa, while leucite-reinforced glass ceramics and feldspathic porcelain have flexural strengths of 160 and 100MPa respectively[1].

Predominantly glassy ceramics

Predominantly glassy ceramics exhibit the excellent optical properties of enamel and dentin, but have limited physical properties. They are composed of three-dimensional networks of atoms having no regular pattern, thus having a structure without form[4]. Glasses in dental ceramics are derived from feldspar, a mined mineral, and are based on silica and alumina[4]. This is where the term feldspathic porcelain is derived[4]. These types of glasses are resistant to crystallization during firing and are biocompatible. Manufacturers use small amounts of filler particles to control effects such as opalescence, color, opacity, and physical properties[2]. These dental ceramics are primarily used to veneer ceramic substructures, inlays, onlays, and veneers[4].

Particle-filled glass ceramics

Particle-filled glass ceramics contain a greater amount of filler particles that enhance the structural properties of the material. Manufacturers add filler particles to the base glass composition to improve mechanical properties such as strength and thermal expansion and contraction[2]. The fillers mostly are crystalline, but can also be composed of higher melting glasses to affect the melting temperature. The particles may be added mechanically during manufacturing or precipitated within the starting glass by special nucleation and growth heating treatments[2]. If the particles are precipitated from the starting glass it is termed a “glass-ceramic”[2]. An example of a glass-ceramic is lithium disilicate crystals.

Non-silica-based Ceramics

Non-silica-based ceramics were developed to address the need of improved fracture strength. They are polycrystalline ceramics that contain little to no glass. These ceramics are tougher and stronger than glass ceramics due to their densely arranged crystalline structure[2]. Aluminum oxide and zirconia oxide serve as reinforcement of the glassy matrix or form a crystalline structure. In general, ceramics consisting of less than 15wt% silica are not regarded as silica-based ceramics. Alumina- or zirconia- oxide forms the matrix in these high strength ceramics[3]. High-strength aluminum-oxide ceramics are indicated for all areas of the mouth for copings and frameworks of full coverage crowns, but are rarely used due to the popularity of higher strength materials. Zirconia is a polymorphic white crystalline material that occurs in three crystallographic forms, monoclinic, tetragonal, and cubic, and has a high flexural strength of around 1000 MPa[1].

Zirconium-Oxide Ceramics

Zirconium-oxide is indicated for conventional and resin-bonded FDPs, full-coverage crowns, and implant abutments. Zirconia has become the predominantly prescribed polycrystalline ceramic. Zirconia's popularity is attributed to its increased mechanical properties and ease of manufacturing[5]. This group of ceramics was developed to address the need of improved fracture strength. They are polycrystalline ceramics that contain no glass. These ceramics are tougher and stronger than glass ceramics due to their densely arranged crystalline structure[2]. Alumina- or zirconia- oxide forms the matrix in these high strength ceramics[3]. Zirconia is a polymorphic white crystalline material that occurs in three crystallographic forms, monoclinic, tetragonal, and cubic, and has a high flexural strength of around 1000 MPa[1]. The monoclinic phase occurs at room temperature to 1170 degrees Celsius. The tetragonal phase is from 1170 to 2370 degrees Celsius, with the cubic phase occurring over 2370 degrees Celsius[5]. Phase transformation exists between the monoclinic and tetragonal phases. Dopants are added to the Zirconia-oxide to stabilize the material. The most common dopant to stabilize zirconia-oxide in the tetragonal phase is yttrium in a 3% molar concentration. The yttria-oxide particles stabilize the zirconia in the tetragonal phase, which limits the phase change from tetragonal to monoclinic. The phase change from tetragonal to monoclinic is undesired but may benefit the restoration during crack formation. Though the fired zirconia in the tetragonal phase is stable, energy exists within the material to cause a phase change to the monoclinic phase; this is known as transformation toughening[4]. The energy at the leading edge of a crack has enough energy to cause the transformation. The transformation to monoclinic has an increase in volume that squeezes the crack closed[4]. This is a beneficial feature of a dental ceramic to reduce crack propagation and failure.

RESIN CERAMIC BONDING

For a tooth treated with a restoration, a strong, durable resin bond provides high retention, improves marginal adaptation, prevents microleakage, and increases fracture resistance[3]. A strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface, which requires roughening and cleaning for adequate surface activation[3]. The strength, bonding protocol, and esthetics differ between the materials and they must be treated correctly. The bonding protocol is different for silica-based glass ceramics and non-silica-based ceramics. Therefore, based on the ceramic material used, a different bonding approach must be utilized. Material choice implies many things about the clinical scenario to include strength of material desired, amount of tooth structure to be removed, esthetic requirements, and the amount of control the clinician exhibits over the gingival environment.

Composite Cements

Resin-based composite cements are currently the recommended material for adhesive luting of ceramic restorations[3]. Resin cements contain inorganic fillers and resin monomers, such as bisphenol A glycidyl methacrylate (BisGMA)/triethylene glycol dimethacrylate (TEGDMA), and urethane dimethyl acrylate(UDMA)[1]. The amount of filler determines the viscosity and flow of the material. Filler-containing composite cements revealed higher bond strengths than resins without fillers, and hybrid composites showed better results than micro-filled resin composites[3]. Highly filled cements may improve abrasion resistance at the marginal area, reduce polymerization shrinkage, and facilitate removal of excess cement. Traditional resin cements do not contain an adhesive functional monomer such as methacryloxydecyl dihydrogen phosphate (MDP)[1]. Cement film thickness has been shown to have an effect on short and long term bond strengths[1].

Resin composite cements can be classified into 3 different groups according to their initiation mode: auto polymerizing, photo activated, or dual-activated[3]. Each type of composite cement has its advantages and disadvantages. Photo activated cements have long handling times and rapid hardening when exposed to light. However, they can only be photo-initiated if light can pass through the ceramic material to an effective depth of cure. Auto polymerizing cements have fixed setting times, and are indicated for opaque materials and high-strength ceramics. Dual-activated cements have extended working times and controlled polymerization. Most dual-activated cements still need to be light cured for final polymerization and hardness.

ADHESIVE CEMENTATION TO SILICA-BASED CERAMICS

Adhesive cementation of silica-based ceramics has been well documented in the literature. The process involves treatment of the intaglio surface, use of a resin cement and tooth treatment that provides optimal bond strength for clinical success. Blatz et al.[3] commented that final adhesive cementation with resin cements increases the fracture resistance of the ceramic material and the abutment tooth. Preferred bonding methods are hydrofluoric acid etching and subsequent silane treatment followed by use of resin cement.

SURFACE TREATMENT OF SILICA-BASED CERAMICS

Surface treatment enhances the interface between the ceramic and cement. This is important to create a strong resin bond by creating micromechanical interlocking and chemical bonding to the ceramic surface[3]. Treatment options include grinding, abrasion with diamond rotary instruments or airborne particles of aluminum oxide, acid etching, or a combination[3]. By applying hydrofluoric acid (HF) solutions of 5% for differing amounts of time, the proper surface texture is achieved through chemical interaction with the silica particles. Lithium-disilicate is etched with 5% HF for 20 seconds, while leucite-reinforced feldspathic porcelain is etched with 5% HF for 60 seconds. After etching of the ceramic surface to provide a substructure for micromechanical retention is completed, additional treatments must be utilized to create the chemical bonding. Utilization of a silane coupling agent has been shown to provide a durable, lasting bond between silica-based ceramics and resin cement.

Silane Coupling Agents

Application of a silane coupling agent is an important step in achieving a significant resin bond. Silanes are organic compounds that contain polymerizable groups, such as methacrylates, at one end and silane alkoxy groups at the other[1]. The methacrylate functional groups can polymerize with an organic matrix of resin materials and the silane alkoxy group can react with the hydroxylated surface[6]. Silanes are bifunctional molecules that bond silicone dioxide with the OH groups on the ceramic surface and a degradable functional group that copolymerizes with the organic matrix of the resin[3]. This provides a chemical covalent (Si-O-Si) and hydrogen bond to the treated ceramic surface. Sorensen et al showed that a combination of ceramic etching and silanization significantly decreased microleakage, which was not achieved by silane treatment alone[3]. Silanization also increases the wettability of the ceramic surface. Studies on the efficacy of silanes after try-in procedures or resilanation of the ceramic restoration show differing results. Residual organic contaminants may decrease bond strength and should be removed before bonding, preferably with phosphoric acids or solvents such as acetone or alcohol[3].

ADHESIVE CEMENTATION TO NON-SILICA-BASED CERAMICS

The preferred protocol for resin bond to zirconia is the combination of airborne particle abrasion to create surface roughness and treatment with a phosphate-containing zirconia primer followed by cementation with an hydrophobic non-phosphate containing resin cement[1]. Kern and Wegner[7] found that phosphate modified BisGMA resin cement on sandblasted ceramic provided the highest bond strengths after 3 and 150 days with medians reported at 47.1-48.8 MPa and 37.4-49.8 MPa respectively.

SURFACE TREATMENT OF NON-SILICA-BASED CERAMICS

Non-silica-based ceramics, and zirconia specifically, do not have the silica molecules that react to the acid etching surface treatments. Different types of surface treatments have been explored for non-silica-based ceramics. They have ranged from airborne particle abrasion, grinding, acidic treatment, laser treatment, and other varied types of surface modification. Most of these provided little to no benefit to bonding. Airborne particle abrasion provided the most improved resin bond, but surface roughness without the application of a phosphate-based primer may not provide a durable resin bond[1]. Phosphate-based primers provide an essential connection to the ceramic material due to the high affinity of the phosphate molecule to the ceramic material.

METHACRYLOXYDECYL DIHYDROGEN PHOSPHATE (MDP) PRIMERS

The chemical structure of organo-phosphate monomer contains polymerizable functional groups and phosphoric acid groups. The polymerizable groups, such as methacrylates, can copolymerize with the matrix of methacrylate based dental resin cements, composites, and adhesives[1].

ZIRCONIUM-OXIDE CERAMICS

Zirconium-oxide is indicated for conventional and resin-bonded FDPs, full-coverage crowns, and implant abutments. Adhesive cementation is not required unless a clinical situation necessitates it, such as a high total occlusal convergence or short abutment teeth. Conventional acid etch has no positive effect on the resin bond to zirconium oxide ceramics[3]. Autopolymerizing resin cement showed the highest bond strength of any surface treatment. Only phosphate-modified resin cement after airborne particle abrasion provided a long-term durable resin bond[3]. MDP has been shown to be hydrolytically stable and does not decrease in bond strength over time.

CONTAMINATION

A good resin-ceramic bond obtained in a strictly controlled clean situation in-vitro might be compromised in clinical situations, leading to a significantly reduced bond[8]. During the try-in procedure of the restoration, contamination of the intaglio surface by saliva, blood or silicone is difficult to avoid[8]. Saliva contamination is frequently one of the main reasons for decreased resin bond strength[8]. Contaminants, such as saliva, blood, and hydrogen peroxide, influenced the bonding between dentin and lithium disilicate ceramics, while desensitizers and disinfectants had no negative effect on the bond strengths[1,9]. Among the different cleaning methods, HF is the most effective in removing contamination with saliva or a silicone disclosing medium[10]. Try-in pastes also prove difficult to remove following their use during try-in[1]. Saliva contamination also significantly affected resin bonds to zirconia and its durability[1].

DETECTING SURFACE CONTAMINANTS

X-ray photoelectron spectroscopy (XPS) is a highly surface sensitive technique for determining the chemical composition of multiphase compounds and for detecting surface contaminants[8]. This technique can be used to determine the presence of saliva contamination on prepared samples.

CLEANING

Cleaning can be accomplished through mechanical and chemical approaches. Scrubbing, abrasion, and grinding can accomplish mechanical cleaning. Chemical cleaning can be accomplished by basic, acidic, neutral, solvent, or emulsion chemicals[8]. Yang et al[8] conducted a study testing the tensile bond strength of zirconia ceramic disks after saliva contamination. The research looked at different cleaning methods, which included rinsing with tap water, immersion in isopropanol alcohol and rinsing with tap water, phosphoric acid etching, airborne particle abrasion, and a control with no saliva contamination. The only method of cleaning that produced a long-term durable bond was airborne particle abrasion.

IVOCLEAN

Ivoclean is a novel ceramic surface treatment to remove contaminants following the try-in procedure. Ivoclean has been recommended for use on glass-ceramics, zirconia, aluminum-oxide, precious metal alloys, base metal alloys, and lab fabricated composite restorations (Ivoclean brochure). Ivoclean is described as effectively cleaning the saliva-contaminated bonding surface, thereby creating a strong, durable bond for adhesive cementation (Ivoclean brochure). Ivoclean is used following the try-in procedure. It is scrubbed onto the intaglio surface and left to react for 20 seconds and thoroughly rinsed with water and dried with oil-free air. The restoration is now ready for adhesive cementation. Ivoclean creates a concentration gradient at the zirconia surface by flooding the area with zirconium oxide particles that attracts the phosphate groups from the some salivary proteins.

TESTING CONDITIONS AND METHODS

Intraoral conditions produce chemical, thermal, and mechanical influences on the ceramic-resin bond[3]. It is necessary to try and replicate these in the laboratory to draw conclusions on the bonds durability. Wegner et al.[11] showed that different storage conditions can affect the tested bonding systems differently regarding the durability of the bond. Long-term water storage and thermocycling of bonded specimens are accepted as ways to simulate aging and to stress the bond interface. Water storage and thermocycling affect the resin itself due to the different coefficient of thermal expansions of the filler particles and surrounding matrix[11]. Significant reduction in bond strength occurs after mechanical cyclic loading.

Preferred bond strength tests are the 3-point bending test, the tensile and micro-tensile test, and the shear and micro-shear test. The most common testing method is the shear bond test. The modified tensile tests may be preferred to eliminate the occurrence of non-uniform interfacial stresses typical to conventional tensile and shear bond tests[3]. The Ultradent shear bond strength testing apparatus is an available system to prepare and test samples for shear bond strength. The Ultradent system provides a standard way to prepare samples by providing a known area for bonding and uniform resin cement addition. The resin piece fits precisely into the crosshead assembly to ensure the force is placed directly on the bonded area and perpendicular to the resin piece.

SUMMARY

Ceramics are becoming an increasingly important part of restorative dentistry due to their esthetics and strength properties. Dental ceramics can be divided broadly into silica-based ceramics and non-silica-based ceramics. Studies have shown that due to the different physical properties, adhesive cementation and surface treatments have to be approached differently. While the differences in adhesive cementation exist there are specific protocols that have been developed for each of the materials to achieve a clinically acceptable bond. Contamination has also proven to be a major issue in reduced bond strength. Care must be taken following contamination from the try-in procedure to clean the ceramic surface to allow for superior bond strength and bond durability. Phosphoric acid has been recommended for cleaning of silica-based ceramics, but has shown to reduce the bond strength of non-silica-based ceramics. Ivoclean is a novel surface treatment developed to remove surface contaminations from zirconia. The effect of Ivoclean on zirconia and silica-based ceramics has not been comprehensively reviewed. Utilizing a single ceramic cleaner would help to simplify and reduce costs associated with the cementation procedure.

PURPOSE

The purpose of this study was to determine the relationship between surfaces that are clean and contaminated (by saliva) then cleaned by different methods and the shear bond strength of different ceramic materials, during accepted bonding protocols. An important goal was to identify if Ivoclean returns zirconia to similar bond strength as an uncontaminated surface. Another goal was to find if Ivoclean is an acceptable product to clean lithium-disilicate restorations after contamination to return the material to acceptable bond strength. The null hypothesis tested was that there would be no difference in shear bond strength of resin cement to zirconia and lithium disilicate ceramics based on the type of cleaning method following saliva contamination, storage and aging.

Chapter 3: Materials and Methods

OVERVIEW

A zirconia (Prettau zirconia; Zirkonzahn GMBH, Italy) blank and lithium disilicate (Emax; Ivoclar Vivadent, Schaan, Liechtenstein) ceramic samples were contaminated with saliva, cleaned, and bonded to a resin cement. Zirconia and lithium-disilicate samples were cleaned with air abrasion, phosphoric acid, water, or Ivoclean. The shear bond strength was tested after 24 hours of storage for the first group of samples. A second set of samples were thermocycled for 10,000 hot-cold cycles to simulate aging and then test for SBS. The results were analyzed statistically to determine if there was a variation from the control bonding strengths.

A power analysis using G*Power for an ANOVA, fixed, special, main effects and interactions, p=0.05, power =80%, 12 groups (6*2), df=5, with an effect size f = 0.35 indicated a total sample size of 111. A group size of 10 (total sample size of 100) was selected. A main effect size of 0.35 is statistically in the medium-large range, but for practical purposes is a sensitive test, equivalent to about 12% of the variance being due to a special effect, and the remainder being due to error variance.

DETAILED METHODOLOGY

Ivoclean (Ivoclar Vivadent, Schaan, Liechtenstein) was tested on two different ceramic materials, zirconia and lithium disilicate ceramic. The specimens were fabricated according to Ultradent's recommended protocols for shear bond strength testing (SBS) and ISO standards, and prepared for cementation. One group (N=10) was used as the untreated control. The other specimens were contaminated in saliva for one minute and subsequently cleaned. One group was cleaned with Ivoclean, one group with phosphoric acid (lithium disilicate) or air abrasion (zirconia), one group with an air/water rinse, and the final group was cemented without cleaning (contamination control). All specimens were bonded utilizing the same resin cement Multilink Automix (Ivoclar Vivadent, Schaan, Liechtenstein). The specimens were stored for 24 hours and then tested for SBS. A second set of specimens were thermocycled for 10,000 hot (55ºC)-cold (15ºC) cycles using a Sabri Enterprises thermocycler (Sabri Dental Enterprises, Inc., Downers Grove, IL), followed by SBS testing on an Instron universal testing machine (E10000, Instron Corporation, Norwood, MA).

Sample Preparation	Lithium Disilicate		Zirconia	
Ivoclean Pilot	Control	Ivoclean Treatment	Control	Ivoclean Treatment
	N=10	N=10	N=10	N=10

Table 1: Ivolclean Pilot Study. The number of samples that were produced for the Ivoclean pilot study which included uncontaminated control samples and uncontaminated Ivoclean treated samples.

Sample Preparation		Lithium Disilicate		Zirconia	
		24 Hour Storage	10,000 Thermocycles	24 Hour Storage	10,000 Thermocycles
Control (No contamination)		N=10	N=10	N=10	N=10
Contaminated Groups	No cleaning	N=10	N=10	N=10	N=10
	Ivoclean	N=10	N=10	N=10	N=10
	Phosphoric Acid	N=10	N=10		
	Air Abrasion			N=10	N-10
	Air/Water	N=10	N=10	N=10	N=10

Table 2: Main experiment sample preparation. This table shows the sample preparation for the samples of the main experiment that were broken down into the control uncontaminated group and the contaminated, not cleaned group. The contaminated, cleaned groups were Ivoclean, Phosphoric acid (lithium disilicate), air abrasion (zirconia), and air/water spray. They were divided to 24 hour storage and 10,000 thermocycles. All groups had 10 samples prepared.

ZIRCONIA SPECIMEN PREPARATION

Zirconia strips (8mm) were sectioned from zirconia blanks (Prettau zirconia; Zirkonzahn USA inc, Norcross, GA), followed by sectioning into 5mm x 8mm x 8mm thick block wafers using a precision saw (Buhler Isomet 5000, Ilinois Tool Works, Lake Bluff, Illinois) with a 0.4 mm thick water lubricated diamond-edge blade. Wafers were dried under a heat lamp for a minimum of 30 minutes and sintered following the manufacturer's instructions in a sintering oven (HT-S speed; Mihm Vogt, Stutensee, Germany) at 1600 degrees Celsius for 8 hrs. The zirconia wafers were secured into an Ultradent mold (Ultradent Corporation, South Jordan, UT), covered with orthodontic resin (Acraweld; Henry Schein, Melville, NY), and cured. The samples were then wet finished to a flat surface with 120, 400, and 600 microns silicon carbide abrasive papers (Coated abrasives; Great Lakes Orthodontics, Tonawanda, NY) on a rotational polisher for 30 seconds per grit. The surface was air abraded with 50 microns alumina oxide for 10 seconds at 15 psi and a distance of approximately 10mm. The specimens were cleaned with a steam cleaner for 20 seconds.

LITHIUM DISILICATE SPECIMEN PREPARATION

Lithium disilicate CAD blocks size C14 (Emax; Ivoclar Vivadent, Schaan, Liechtenstein) were sectioned using a linear precision saw into 5 mm thick block wafers and crystallized following the manufacturers instructions in a Programat EP 5010 (Ivoclar Vivadent, Schaan, Liechtenstein) furnace. The lithium disilicate wafers were secured into an Ultradent mold (Ultradent Corporation, South Jordan, UT), covered with orthodontic resin (Acraweld; Henry Schein, Melville, NY), and cured. The samples were then wet finished to a flat surface with 120, 400, and 600 micron silicon carbide abrasive papers (Coated abrasives; Great Lakes Orthodontics, Tonawanda, NY) on a rotational polisher for 30 seconds with each grit. The surface was etched with 5% hydrofluoric acid (IPS Ceramic Etching Gel; Ivoclar Vivadent, Schaan, Liechtenstein). The specimens were cleaned with a steam cleaner for 20 seconds.

ULTRADENT SAMPLE PREPARATION

An Ultradent shear bond testing apparatus was used to prepare the ceramic samples for shear testing. The sample preparation consisted of a mold that prepared 15 cylinders measuring 1 inch diameter by 1 inch in length. Tape was placed over one side to secure the ceramic samples. The samples were secured to the tape. Orthodontic resin was mixed and poured into the mold covering the ceramic samples. The mold was placed into a water bath to finish curing once the initial set occurred. Samples were removed from the mold after curing was completed. The samples were then ground to remove the outer surface that created a uniform, parallel surface for bonding using the Ultradent grinding mandrel. The grinding mandrel produced a random grinding pattern on the bonding surface by rotating the sample during grinding. Following grinding, the samples were ready for their surface preparation and bonding procedures. The bonding procedures utilized the Ultradent bonding clamp and bonding mold inserts. The clamps stabilized the samples and allowed precise contact of the bonding mold inserts. Ultradent’s bonding mold inserts created intimate contact and a known sample-bonding surface of 2.38 mm diameter.

PILOT IVOCLEAN UNCONTAMINATED CONTROL

Twenty zirconia samples and 20 lithium disilicate samples were prepared and assigned to a control group (N=10) and an Ivoclean group (N=10). The control group was not cleaned with any material. The Ivoclean group was scrubbed with Ivoclean for 60 seconds and rinsed with air-water spray for 60 seconds. Following sample preparation, the zirconia and lithium disilicate control groups were scrubbed and silane (Monobond Plus; Ivoclar Vivadent, Schaan, Liechtenstein) was applied on the bonding surface for 60 seconds and dried. The samples were then ready for the bonding with a resin cement (Multilink Automix; Ivoclar Vivadent, Schaan, Liechtenstein).

UNCONTAMINATED CONTROL SAMPLES

Twenty zirconia samples and 20 lithium disilicate samples were prepared. Following sample preparation, the zirconia and lithium disilicate samples were scrubbed with Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) on the bonding surface for 60 seconds and dried. The samples were then bonded with a resin cement (Multilink Automix; Ivoclar Vivadent, Schaan, Liechtenstein). The prepared zirconia and lithium disilicate samples were assigned to a 24 hr storage group (N=10) and a thermocycled group (N=10) for each material.

CONTAMINATION

The contamination procedure consisted of collecting an adequate sample of saliva from one volunteer under an IRB approved protocol (reference number C.2016.152n). The saliva was collected after the volunteer fasted from food and liquid for two hours prior to sample collection. The different ceramic samples were scrubbed with the saliva using a microbrush for one minute to simulate try-in contamination.

BONDING PROCEDURE

The prepared ceramic samples in orthodontic resin were placed into the Ultradent bonding clamp to secure the samples to the bonding mold insert. A dual-cure resin cement was mixed and injected into the Ultradent bonding mold insert to a height of approximately 3 mm and cured for 20 seconds (bluphase G2 light curing unit; Ivoclar Vivadent, Schaan, Liechtenstein). The samples were removed from the Ultradent jig and ready for SBS testing.

CONTAMINATED CONTROL SAMPLES

Twenty zirconia samples and 20 lithium disilicate samples were prepared. Following sample preparation, the zirconia and lithium disilicate control groups were contaminated following the procedure previously described. The samples were air-dried and scrubbed with Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) on the bonding surface for 60 seconds and dried. The samples were bonded with a resin cement (Multilink Automix; Ivoclar Vivadent, Schaan, Liechtenstein). The prepared zirconia and lithium disilicate samples were assigned to a 24 hr storage group (N=10) and a thermocycled group (N=10) for each material.

IVOCLEAN CLEANED SAMPLES

Twenty zirconia samples and 20 lithium disilicate samples were prepared. Following sample preparation, the zirconia and lithium disilicate control groups were contaminated following the procedure previously described. The samples were subsequently cleaned with Ivoclean by scrubbing for 20 seconds and allowing the solution to react for 40 seconds. The samples were rinsed with air/water spray to remove the Ivoclean for 20 seconds. The samples were then treated with Monobond Plus (Ivoclar

Vivadent, Schaan, Liechtenstein) on the bonding surface for 60 seconds and dried. The samples were bonded with a resin cement (Multilink Automix; Ivoclar Vivadent, Schaan, Liechtenstein). The prepared zirconia and lithium disilicate samples were assigned to a 24 hour storage group (N=10) and a thermocycled group (N=10) for each material.

PHOSPHORIC ACID AND AIR ABRASION CLEANED SAMPLES

Twenty zirconia samples and 20 lithium disilicate samples were prepared. Following sample preparation, the zirconia and lithium disilicate control groups were contaminated following the procedure previously described. The zirconia samples were cleaned with air abrasion with 50 micron alumina oxide for 10 seconds at 15 psi and a distance of approximately 10mm. The lithium disilicate samples were cleaned with 37% phosphoric acid (Gel Etchant; Kerr Corporation, Orange, CA) for 60 seconds and rinsed with air/water spray for 20 seconds. The samples were air-dried and scrubbed with Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) on the bonding surface for 60 seconds and dried. The samples were bonded with a resin cement (Multilink Automix; Ivoclar Vivadent, Schaan, Liechtenstein). The prepared zirconia and lithium disilicate samples were assigned to a 24 hour storage group (N=10) and a thermocycled group (N=10) for each material.

AIR/WATER SPRAY CLEANED SAMPLES

Twenty zirconia samples and 20 lithium disilicate samples were prepared. Following sample preparation, the zirconia and lithium disilicate control groups were contaminated following the procedure previously described. The samples were subsequently cleaned with air/water spray for 20 seconds. The samples were dried and treated with Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) on the bonding surface for 60 seconds and dried. The samples were bonded with a resin cement (Multilink Automix; Ivoclar Vivadent, Schaan, Liechtenstein). The prepared zirconia and lithium disilicate samples were assigned to a 24 hour storage group (N=10) and a thermocycled group (N=10) for each material.

BOND STRENGTH TESTING

The specimens were stored at room temperature for 24 hours. The stored samples were then loaded perpendicularly using the Ultradent test base clamp in a universal testing machine (Instron E10000, Instron Corporation, Norwood, MA). The samples were loaded by shear force using Ultradent's crosshead assembly at a rate of 1mm/min until failure. Shear bond strength values were recorded in N at the peak load of failure and calculated to MPa by dividing by the surface area of the samples.

DATA ANALYSIS

Descriptive and inferential statistics were performed to analyze the collected data. The descriptive statistics looked at the normality of the distribution and presence of outliers. Shapiro-Wilk normality test, D'Agostino-Pearson omnibus test, ROUT test for outliers, and Student's t-test were used. Inferential statistics compared the results from the different groups to see if there was a correlation from the different variable tested. One and two-way ANOVA were used to analyze the data, with p values less than 0.05 being taken as significant. Tukey's post-hoc test was used if a significant ANOVA result were found.

Chapter 4: Results

Two different ceramic samples were tested to determine the effect on bond strength of Ivoclean application, contamination, and four different cleaning methods. Results were recorded in Newtons on the Instron universal testing machine and converted to megapascals (MPa) for data analysis. Conversion was performed using the following equation MPa= X N/4.33 mm^2, where X represents the Newtons recorded divided by the area of the bonded samples.

IVOCLEAN PILOT STUDY

A pilot study was performed to determine the effect of Ivoclean on uncontaminated samples (Figure 1). Lithium disilicate control samples (n=9) gave a mean shear bond strength of 24.66 ± 5.00 (SD) MPa. The samples (n=10) treated with Ivoclean recorded a mean SBS of 24.83 ± 5.77 (SD) MPa. The Ivoclean treated group had one outlier that was identified by the ROUT test for outliers (Q=5%). Following the removal of the outlier, the Ivoclean treated samples passed for normality. Since parametric tests are relatively robust towards modest deviations from normality, and this was only one sample that was borderline (Q=5% is relatively relaxed filtering), all samples were retained for analysis.

A two-tailed unpaired t-test was performed to compare the control and Ivoclean-treated bond strengths for lithium disilicate. There was no significant difference (p=0.95) between the lithium disilicate uncontaminated control and the Ivoclean treated uncontaminated control.

Zirconia control samples (N=10) gave a mean SBS of 18.87 ± 4.54 (SD) MPa. The zirconia samples (N=10) treated with Ivoclean gave a mean SBS of 18.59 ± 3.12

(SD) MPa. These samples passed both tests for normality ($p>0.71$). Zirconia showed no significant difference (t-test; $p=0.88$).

For both lithium disilicate and zirconia, the control and Ivoclean samples tested had a statistically similar SBS that produced a normal distribution. With the samples being statistically similar, no effect could be identified from treatment with Ivoclean on uncontaminated surfaces.

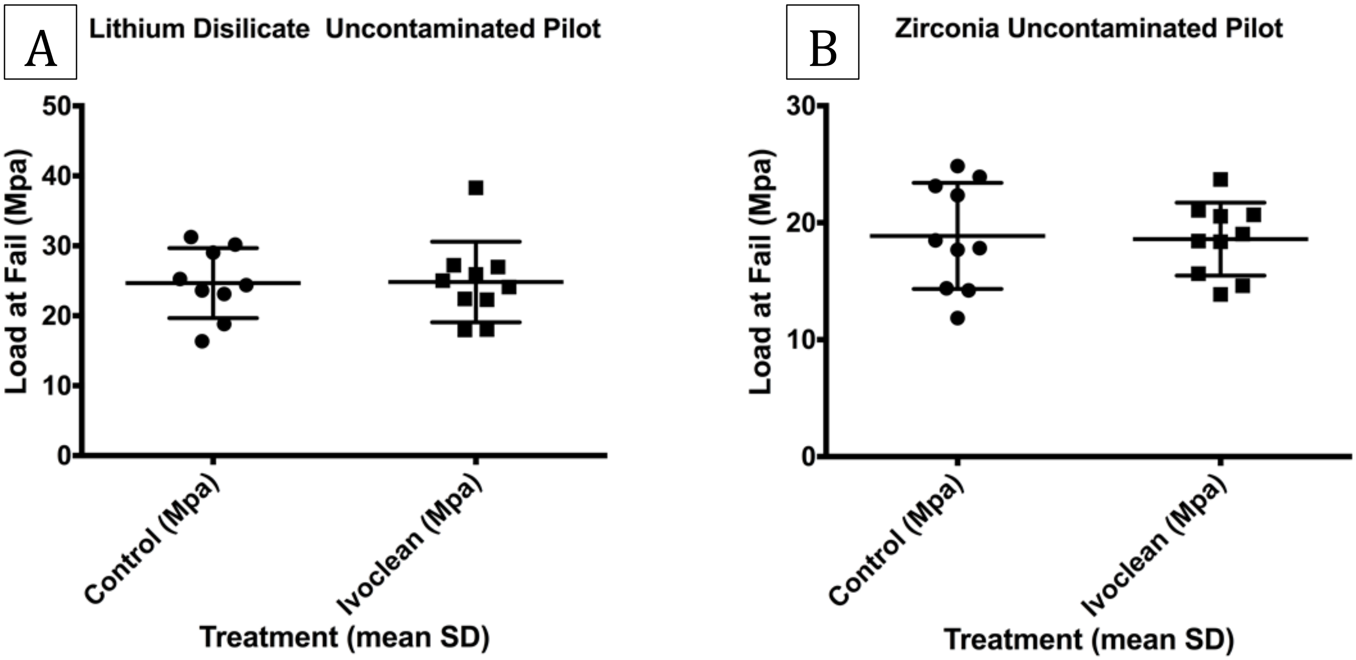

Figure 1: Distribution data for Load at Failure for Lithium Disilicate (A) and Zirconia (B) treated or untreated with Ivoclean. The mean (large bar) and standard deviation (smaller bars) are shown.

MAIN EXPERIMENT

The results from the main experiment are listed in Table 3. Table 3 shows the means, standard deviations, and the number of samples that were tested for SBS. No spontaneous debonding occurred for the 24 hour water storage samples. Table 1b catalogs significant spontaneous debonding of the thermocycled samples resulting in a reduced number of samples for SBS testing.

A	Untreated control	Contaminated	Ivoclean	Phsophoric acid	Air abrasion	Air/water
Lithium Disilicate	**31.41±8.23** **N=10**	**18.15±9.29** **N=10**	**22.83±5.58** **N=10**	**23.04±9.15** **N=10**		**22.36±6.05** **N=10**
Zirconia	**23.92±4.17** **N=10**	**14.75±3.99** **N=10**	**17.29±4.59** **N=10**		**20.63±8.51** **N=10**	**14.34±3.97** **N=10**

B	Untreated control	Contaminated	Ivoclean	Phosphoric acid	Air abrasion	Air/water
Lithium Disilicate	**23.48±4.00** **N=9**	**8.17±4.03** **N=6**	**8.93±3.15** **N=4**	**25.83±5.00** **N=4**		**14.79±2.44** **N=6**
Zirconia	**15.93±3.96** **N=10**	**8.00±1.87** **N=5**	**N=0**		**14.99±3.28** **N=9**	**11.00±3.81** **N=10**

Table 3: Means and standard deviation values for shear bond strength tests after 24 hrs (A) and 10000 thermocycles (B). Values are in MPa. The numbers of samples that were examined for shear bond strength (i.e., for post-thermocycling, the numbers that did not de-bond during the cycling process) are shown as N= value.

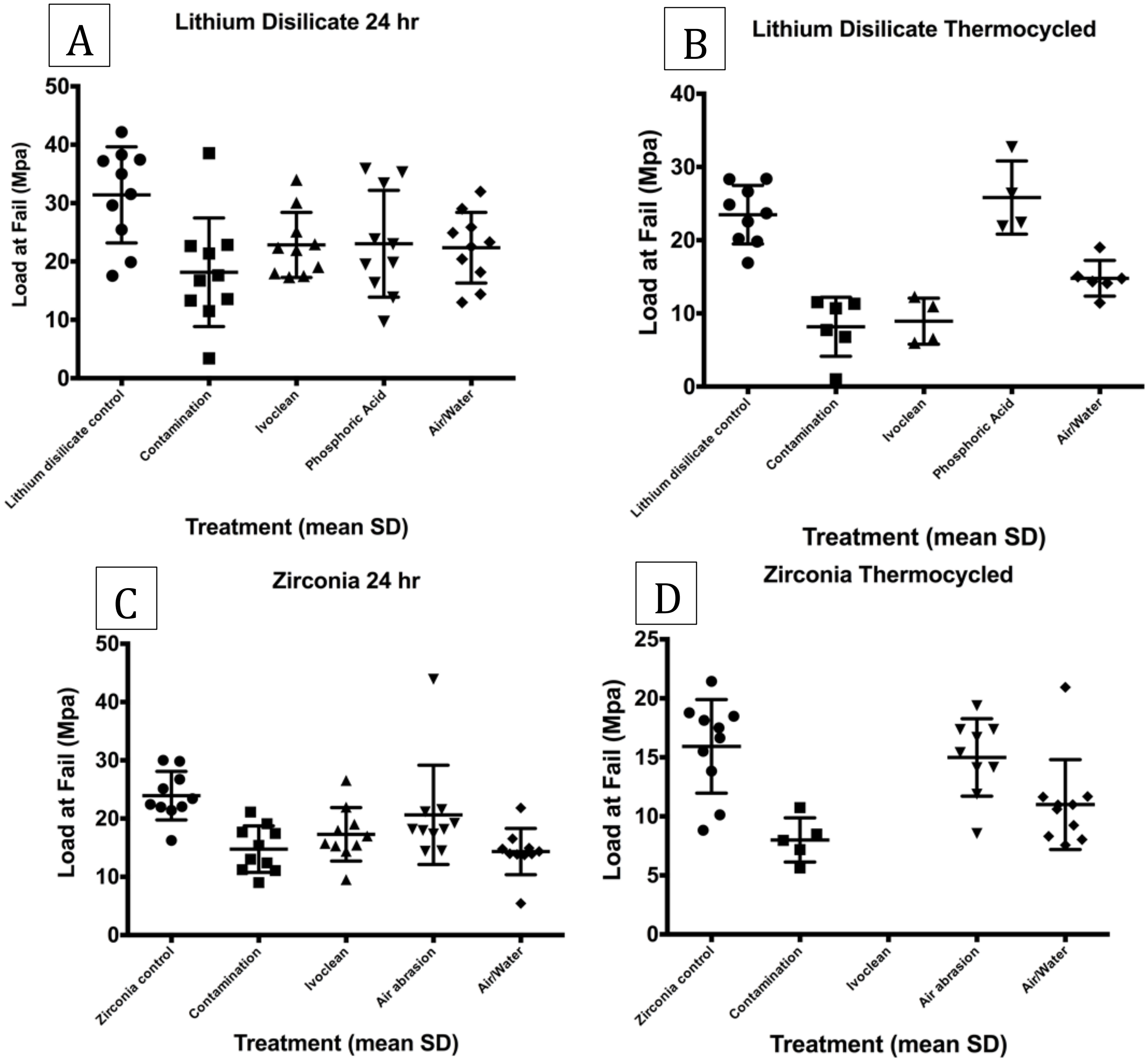

Figure 2: Distribution data for SBS with and without thermal cycling. Scatter plots are shown for: A; Lithium Disilicate 24hr, B; Lithium Disilicate thermocycled; C; Zirconia 24 hr; D Zirconia thermocycled. The mean (large bar) and standard deviation (smaller bars) are shown.

Figure 2 shows the distribution of the failure rates of the tested samples broken down by individual groups tested. Large bars show mean, small bars standard deviation.

LITHIUM DISILICATE SAMPLES

Figure 2A exhibits a uniform distribution for each of the five test groups in the 24 hour storage samples with no obvious outliers; this was confirmed by tests for normality (p>0.15). The number of debonded samples was significant in the thermocycled group and only the control group was tested for normality (the other 4 groups had too few samples to test). The distribution was uniform for all five test groups as visualized by the graphs in Figure 2. The range of standard deviations between the groups was less than 4, and both sets passed the Brown-Forsythe and Bartlett's tests for homogeneity of variance (p>0.46). Homogeneity of variance was assumed.

ZIRCONIA SAMPLES

The zirconia group's data had relatively uniform distributions as visualized on the scatter plots in Figure 2C and Figure 2D, except for a remarkably high value in the 24 hour air abrasion group. The control, contaminated and Ivoclean groups all passed both tests for normality (p>0.39). Outliers were identified in the 24 hour air abrasion (highest value) and 24 hour air/water groups (highest and lowest values) using the ROUT test (using a conservative Q=1%). After removal of these outliers, the air abrasion group passed both tests for normality, but the air/water group still failed both, with high positive kurtosis and skew. Since ANOVA is relatively robust to deviations from normality, normality was assumed with all values included.

All samples debonded in the Ivoclean group after being thermocycled, and therefore Ivoclean could not be tested. One outlier was identified using the ROUT test in the air/water group and after removal, all groups passed both tests for normality. Normality was assumed with all values included.

For the zirconia pre- and post-thermocycling datasets, neither the Brown-Forsythe test nor Bartlett's test detected heterogeneity of variance.

EFFECT OF SURFACE CONTAMINATION AND CLEANING MATERIALS ON LITHIUM DISILICAATE SBS

Surface contamination and cleaning methods contribute to effects on bond strength. Two-way ANOVA was used to compare contamination and cleaning (five levels), and thermocycling (two levels) on SBS of lithium disilicate. No significant interaction was detected between contamination and cleaning, and heat treatment (p=0.052). Contamination and cleaning (p<0.0001) and thermocycling (p<0.0001) each had a significant effect on the SBS of lithium disilicate. Broadly, thermocycling showed a trend for a reduction in the SBS in comparison to 24 hour, and contamination with saliva reduced the immediate and post-thermocycling bond strengths. Phosphoric acid showed a different pattern with respect to thermocycling that air/water or Ivoclean.

Pre-thermocycling, saliva contamination showed a significant decrease in SBS in comparison to the untreated control (18.2 vs 31.4 MPa, p=0.0003). Ivoclean treatment of saliva contaminated lithium disilicate showed a significant difference to the untreated control (22.8 vs 31.4 MPa, p=0.043), but was not significantly different from the saliva contaminated SBS (22.8 vs 18.2MPa, p=0.53). Therefore, Ivoclean treatment of saliva contaminated lithium disilicate resulted in no significant improvement in the SBS, which failed to reach the uncontaminated control levels. Air/water treatment was also significantly lower than the untreated control (22.4 vs 31.4 MPa, p=0.028), and was not significantly different from the saliva contaminated SBS (p=0.63).

In contrast, at 24 hours phosphoric acid cleaning gave an SBS not significantly different from the untreated control (23.0 vs 31.4MPa, p=0.051), although also not significantly different from the saliva contaminated SBS (p=0.48). All other pairwise comparisons were not significant (p=1.0).

Following thermocycling of lithium disilicate there were no significant differences in the SBS of the untreated control (p=0.059), phosphoric acid (p=0.96) and air/water (p=0.150) groups in comparison to the 24 hour values. That is, the untreated control, phosphoric acid, and air/water were able to withstand thermocycling without a significant decrease in the SBS. In contrast, the saliva contamination group showed a significant decrease in SBS (from 18.2 to 8.2MPa, p=0.026), as did the Ivoclean group (from 22.8 to 8.9MPa, p=0.004). All other comparisons showed no significant difference (p>0.059).

Post-thermocycling, saliva contamination showed a significant decrease in SBS in comparison to the untreated control (8.2 vs 23.5 MPa, p=0.0005). Ivoclean retained a significant difference to the untreated control post-thermocycling (8.9 vs 23.5MPa, p=0.005), and was not significantly different to the saliva contaminated surface (p=1.0). Air/water cleaning (18.6 MPa) was not significantly different from either untreated control (p=0.11), or from saliva contaminated (p=0.43). Phosphoric acid cleaning gave a SBS (25.8MPa) that was not significantly different than the untreated control (p=0.98), and significantly higher than the contaminated surface (p=0.001), as well as the Ivoclean treated surface (p=0.006). All other pairwise comparisons were not significantly different (p>0.09). Phosphoric acid cleaning was able to maintain a higher SBS than other cleaning methods but was not statistically different from air/water cleaning. Collectively, saliva contamination caused an initial decrease in bond strength, and thermocycling lead to a further decrease not seen with the uncontaminated surface. Without removing saliva, the effects of thermocycling were more pronounced and statistically Ivoclean did not show a beneficial cleaning effect.

EFFECT OF SURFACE CONTAMINATION AND CLEANING MATERIALS ON ZIRCONIA SBS

Two-way ANOVA was used to examine the effect of all cleaning methods except Ivoclean (which gave no remaining samples post-thermocycling) and the effects of thermocycling. There was no significant interaction between contamination and cleaning and heat treatment (p=0.465). However, contamination and cleaning (p<0.0001) and thermocycling (p<0.0001) each had a significant effect on the SBS of Zirconia, and acted independently. Figure 3b shows the mean values plotted for the pre- and post-thermocycled zirconia groups and visually illustrates the trend of thermocycling to reduce SBS. It is also evident that contamination with saliva reduced the immediate and post-thermocycling bond strengths.

Pre-thermocycling, saliva contamination showed a significant decrease in SBS in comparison to the untreated control (14.8 vs 23.9 MPa, p=0.0003). Air/water treatment (14.3MPa) was also significantly lower than the untreated control (p=0.0001), and was not significantly different from the saliva contaminated SBS (p=1.0).

In contrast, air abrasion cleaning gave an SBS (20.6MPa) not significantly different from the untreated control (p=0.41), and significantly higher than the saliva contaminated SBS (p=0.034), or that of the air/water cleaned surface (p=0.020). All other pairwise comparisons were not significant (p=1.0).

Comparison of treatment means before and after thermocycling showed a significant decrease in the untreated control (23.9 vs 15.9MPa, p=0.001) and the saliva contaminated (14.8 vs 8.0MPa, p=0.044) groups, and in the air abrasion cleaned group (20.63 vs 15.0 MPa, p=0.044). In contrast, the air/water treatment did not show a significant difference (14.3 vs 11.0MPa, p=0.4).

Post-thermocycling, saliva contamination showed a significant decrease in SBS in comparison to the untreated control (8.0 vs 15.9 MPa, p=0.016). Now, air/water cleaning (11.0 MPa) was not significantly different from either untreated control (p=0.10), or from saliva contaminated zirconia (p=1.0).

Post-thermocycling, air abrasion cleaning gave a SBS (15.0MPa) that was still not significantly different than the untreated control (p=0.97), and significantly higher than the contaminated surface (p=0.047). All other pairwise comparisons were not significantly different (p>0.26).

Collectively, saliva contamination decreased the SBS for Zirconia, and thermocycling further decreases the bond strength. Air abrasion appeared to be the best cleaning method for zirconia, giving no significant difference to the control pre- or post-thermocycling.

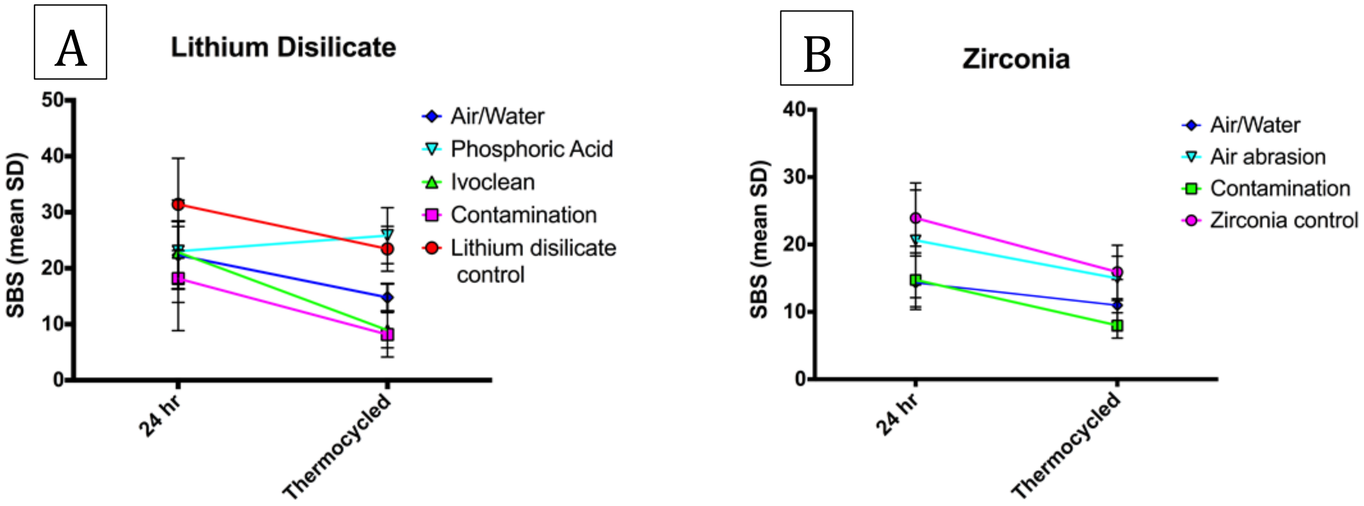

Figure 3: Two-way ANOVA analysis of the effects on contamination and cleaning and of thermocycling on SBS. A: Lithium Disilicate; B: Zirconia. The bars show standard deviation.

For zirconia, one-way ANOVA was used to examine the effects of cleaning methods pre-thermocycling. A highly significant difference was found between the treatments (p=0.0007). Tukey's post hoc analysis showed that saliva contamination significantly lowered the SBS (14.8 vs 23.9 MPa, p=0.003). The Ivoclean treated surface (17.3MPa) was not significantly different from either the untreated control (p=0.058) or the saliva contaminated surface (p=0.82). The air/water cleaned surface (14.3MPa) was significantly lower than the untreated control (p=0.002), and not significantly higher than the saliva contaminated surface (p=1.0). All other pairwise comparisons showed no significant differences (p>0.12). At 24 hours, saliva contamination and air/water spray showed significant reduction in SBS and were significantly different from the uncontaminated controls. While those groups showed marked reduction, they were not significantly different from the other cleaning methods. Therefore, Ivoclean appeared to show a trend to modest benefit, but did not reach a level of statistical significance.

FAILURE OF SAMPLES DURING THERMOCYCLING

Failure of samples during thermocycling was examined using Fisher's exact test. For lithium disilicate, the proportions did not differ significantly from chance (p=0.142). However, for zirconia, a significant difference in the proportion of failed samples was detected (p<0.0001). Post hoc analysis by pairwise chi-square tests was used to compare the contaminated control and cleaning methods to the uncontaminated control (10 pass/0 fail) with a Bonferroni correction to alpha for multiple comparisons (n=4; corrected alpha=0.0125). The Ivoclean group showed a significant difference (0 pass/10 fail; p<0.001), as did the saliva-contaminated group (5 pass/5 fail; p=0.010). That is, the contaminated (5 pass/5 fail) and Ivoclean (0 pass/10 fail) zirconia samples showed a significantly higher failure rate compared to the other cleaning methods for zirconia. Ivoclean use on lithium disilicate (4 pass/6 fail) did not significantly differ from other groups. Thermocycling is identified as a reason for bond reduction and failure.

Chapter 5: Discussion

Bond strengths of zirconia and lithium disilicate ceramic materials have been studied extensively. Contamination, cleaning methods, and aging (thermocycling) have all been shown to alter bond strengths independently of one another. This research examined the effect of these three factors to determine if specific cleaning methods were able to restore initial uncontaminated SBS after short term storage and following 10000 thermocycles.

CONTAMINATION

Ideally, uncontaminated samples would be used for bonding in the patient, but contamination occurs during try-in procedures that can have a significant adverse impact on bond strength. In the present study, contaminated samples without cleaning produced the lowest mean SBS, 18.15 MPa for 24 hour stored lithium disilicate and 14.75 MPa for stored zirconia, and only 8.17 MPa for thermocycled lithium disilicate, and 8.00 MPa for thermocycled zirconia samples. Clearly a contaminated surface cannot provide adequate bond strength. This is likely due to the salivary proteins affinity to the glass substrate that reduces the ability of silanes to make intimate contact with the ceramic. Modern silanes contain MDP, which is critical in the success of bonding due to its affinity for the ceramic and resin cement. Applying a MDP primer as the last step can aid in improving the bond. Contamination inhibits the bond and should be cleaned prior to the bonding procedure to prevent the decrease in restoration longevity. The contamination decreases the wettability of the ceramic materials inhibiting the silane from wetting the ceramic surface effectively. The reduced wettability would lead to the decrease in SBS. Isolation and control of bonding procedures is paramount in the reduction of contamination.

According to this research, no cleaning methods reached the SBS of the uncontaminated control. SBS was the only metric used in this study to determine the quality of the bond or lack thereof, with the assumption this was related to removal of contamination. Other methods such as XPS could be utilized to measure the amount and significance of contamination to quantify the amount of contamination to reduced SBS. Reduction of SBS from contamination was seen in both stored and aged groups suggesting that the contamination decreases initial SBS and contributes to degradation of bond from aging.

AGING (THERMOCYCLING)

Aging occurs after a restoration has been bonded intraorally. Thermocycling has been used as an acceptable way to simulate aging and has been shown to decrease bond strength. Aging significantly affected the bond strengths over all of the test groups in this study. Uncontaminated lithium disilicate decreased from 31.41 to 23.48 MPa in the thermocycled group and the control zirconia decreased from 23.92 to 15.93 MPa. Thus, aging contributes significantly to the decrease in bond strength over all.

Spontaneous debonding also occurred during thermocycling. This was significant and was particularly pronounced in the Ivoclean test groups, where the thermocycled Ivoclean zirconia samples all debonded, while 6/10 debonded from the thermocycled Ivoclean lithium disilicate test group. Interestingly, the thermocycled uncontaminated samples only had one lithium disilicate sample spontaneously debond. This illustrates two things. The first was that thermocycling reduced bond strength. The second was that cleaning methods do not completely rid the contamination from the ceramic structures that caused reduced bond strength initially.

Thermocycling requires the samples be loaded into an apparatus that moves the samples from a warm water bath to a cold-water bath. The motion of the arm, basket, and samples within the basket may cause added stress, which may have aided in debonding samples that already had a reduced bond strength. The lithium disilicate samples were generally affected by spontaneous debonding, while the contaminated and Ivoclean zirconia samples were affected the most.

CLEANING

Various cleaning methods have been proposed to reduce the effect of contamination: Ivoclean, phosphoric acid for lithium disilicate, air abrasion for zirconia, and air/water spray. Initially, cleaning methods maintain an adequate bond strength following 24 hours of storage in water but do not return contaminated samples to the uncontaminated bond strength. Collectively, pre-thermocycling, neither Ivoclean nor air/water treatment showed any benefit for amelioration of the adverse effects of saliva contamination on SBS for lithium disilicate. Phosphoric acid appeared to show a modest benefit.

Short-term results showed that the cleaned samples were not statistically significant from the uncontaminated control, but after 10000 thermocycles, the cleaning methods were not the same. In the lithium disilicate group, phosphoric acid performed the best at maintaining a bond strength similar to the uncontaminated samples. Ivoclean and air/water spray did not achieve results statistically similar to the untreated control.

Air abrasion cleaning produced the best results for zirconia following thermocycling with a mean of 14.99 MPa. Thermocycled samples cleaned with Ivoclean produced no results due to complete spontaneous debonding during the aging process.

Air/water spray also produced results not significantly different from the uncontaminated control after thermocycling.

While Ivoclean did not manage to reproduce initial bond strengths, Ivoclean used on uncontaminated ceramic samples and stored short term did not produce any significant effects on SBS. Providing long term storage and thermocycling on uncontaminated samples treated with Ivoclean may elicit different results to help explain the debonding of samples treated with Ivoclean due to the effects of the Ivoclean treatment.

DENTAL MATERIALS

Differences in testing conditions contribute to potentially different results from study to study. Standardization amongst material properties could be inconsistent between different studies. The composition, shrinkage, crystallization profile, and strengths can vary amongst manufacturers, especially for zirconia products. The other studies mentioned in the comparison to other studies section below utilize a different zirconia manufacturer. The different zirconia profiles might react differently to saliva contamination, cleaning methods, and storage/aging conditions. Therefore, Ivoclean may have a different effect on different brands of zirconia. Thermocycling might also affect the different materials to a differing effect. The size, shape, and spacing of the zirconia crystal could expand and contract at different rates. Zirconia with more translucency or less flexural strength could behave differently to the aging procedures. Different expansion and contraction profiles between the resin and ceramic materials results in stress on the bond that in turn diminishes the bond strength. Crystallization and grain boundaries are important factors that regulate translucency and strength. Different zirconia materials have similar structural profiles, but can vary in the their translucency and physical properties.

LIMITATIONS TO THIS STUDY

Limitations with respect to this study can be broadly categorized as sample preparation and testing. Sample preparation relies on the properties of the materials being used and their sensitivity to handling. Testing requires a consistent method and contact with the sample to elicit force on the same spot of the samples. Errors may be compounded throughout the preparation and testing steps.

Samples were prepared following a method proposed by Ultradent to test adhesives for SBS. The method aims to minimize the variance in samples by providing a systematic approach to sample preparation. However, variations still exist in this process and cannot be quantified without examination of each sample at each step of the procedure. One critical factor is the parallel sides that are meant to be perpendicular to the testing apparatus. Flexure was noticed during grinding while using the Ultradent grinding assembly. If the sides did not remain parallel, then other forces could be placed on the sample that would affect the data collection.

Another limitation was that the saliva contamination of the materials was not quantified. Quantifying the amount of contamination by XPS would have shown if samples were uniformly contaminated. If the contamination was not uniform SBS could have been different during testing.

Thermocycling potentially created several errors due to the force at which the samples were transferred between water baths. This may have created stress on the bonding interface due to agitation between samples that contributed to samples debonding. The water bath temperatures were also not fully controlled at the specified temperatures. Especially, the cold-water bath was not able to maintain the prescribed temperature of 5 degrees Celsius. This was due to the large bulk of samples carrying warm water to the bath and the cooling pump not being adequate. However, despite the temperature differential not being as great as recommended for testing, a clear impact of thermocycling on bond strength was observed.

Loading samples onto the Instron universal testing machine requires a precise contact. If the sample surface is not parallel to the crosshead assembly, the sample will not be perpendicular to the force applied. Torque and off axis forces may change the failure mode of the tested samples. This could explain some of the few unusually high values (outliers) that were detected statistically.

COMPARISON TO OTHER STUDIES

The results from this study report similar mean SBS values as other studies, but differ in regards to the results of Ivoclean. One study showed that uncontaminated lithium disilcate had an SBS of 29.7 (5.9) MPa, Ivoclean treatment 30.1(6.0) MPa, phosphoric acid 25.0(8.5) MPa, air/water 17.6 (8.4) MPa, and uncleaned 7.8 (2.5) MPa (Alfaro, 2016; values in parentheses are standard deviations). While this study by Alfaro et al shows results that are comparable amongst the different methods, the samples were not aged to determine if the SBS deteriorated during thermocycling. Versa-link silane and NX3 resin cement was used to bond the samples. The difference in cementation materials and lack of aging limits the comparison of the most significant deterioration of SBS during thermocycling.

Another study by Angkasith et al.[21] looked at the effect of cleaning methods and point of MDP application on contamination of zirconia. Their results showed that MDP as the final step was important to maintaining higher bond strengths on zirconia. The SBS in this study were control 25.8 (6.1) MPa. The saliva contaminated samples cleaned by air abrasion followed by MDP gave 23.1 (1.7) MPa, water followed by MDP 10.4 (3.6) MPa, phosphoric acid followed MDP 9.9 (2.0) MPa, and Ivoclean followed by MDP 22.7 (3.8) MPa. The study also showed that MDP treatment followed by saliva then water produced SBS of 20.6 (6.3) MPa. They found that the MDP/saliva/water, saliva/air abrasion/MDP, and saliva/Ivoclean/MDP were not statistically different from the uncontaminated control[21]. All of the samples were thermocycled in this study, so the decrease from contamination alone cannot be identified. Also, the effect of thermocycling cannot be quantified beyond the groups tested due to not maintaining an initial test group. Both of these studies show Ivoclean maintained SBS similar to the control with the two materials tested in this study.

Comparing the previous two studies to the results of this study, similarities are realized in some respects. The lithium disilicate study by Alfaro et al.[22] maintained similar uncontaminated control values (31.41 vs 29.7 (Alfaro)), phosphoric acid (23.04 vs 25.0[22]), and air/water spray (22.36 vs 17.6 (Alfaro)). The contaminated but not cleaned and Ivoclean samples varied between the two studies. Thermocycling was not performed during the Alfaro et al study and cannot be compared to the results of thermocycling in this study.

Zirconia was examined in the Angkasith et al.[21] study and determined that MDP as the last step was important for an elevated SBS. Monobond plus was used as the final surface treatment prior to bonding in this current study. The SBS of the current study were lower than the mentioned study following thermocycling. Ivoclean could not be compared due to the debonding of all the Ivoclean samples during thermocycling in the current study.

As mentioned previously, the ceramic material may be a major factor in the reaction to contaminants, cleaning methods, and silane. Aging effects might interact with the two materials differently. Specifically, different brands and physical properties of zirconia might produce different SBS results. Direct comparison of different ceramic materials are limited if these factors strongly influence SBS. Further studies will be needed to compare different manufacturers of zirconia and aging methods on the SBS of the bonded samples. Looking more in depth at the interaction of saliva to ceramics is imperative to knowing the exact mechanism of cleaning and if there are cleaning methods that are more in tuned to effectively removing contaminants. Cleaning methods outside the field of dentistry should be examined to see the most effective method for removing contaminants.

Chapter 6: Conclusion

The conclusions that can be gained from this research are as follows:

1. Saliva contamination and aging (thermocycling) decrease bond strengths independently from each other and compound one another.
2. Cleaning methods in this study generally do not return bond strengths to that of the uncontaminated samples. Therefore the null hypothesis was rejected.
3. Evidence was not found to support the use of Ivoclean for either zirconia or lithium disilicate, although there was a trend to a modest benefit with zirconia. Further research is indicated to determine if Ivoclean is an effective cleaning protocol.
4. Air abrasion for zirconia and phosphoric acid for lithium disilicate maintained bond strengths not significantly different from the uncontaminated samples following thermocycling.

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