EFFECTIVENESS OF UNIVERSAL ADHESIVE BONDING AGENTS
ON THE SHEAR BOND STRENGTH TO
LITHIUM DISILICATE
CERAMICS

by

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DEDICATION

This thesis is dedicated to my mother, my wife, and my son for their support, love, and patience, which were my inspiration for success.

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INTRODUCTION

Dental adhesives play an important role in dental treatment as they establish an effective bond with the tooth structures. Restoration retention depends on adhesives' complex physical and chemical properties. Therefore, a good understanding of the composition, characteristics, and mechanisms of such adhesive systems are essential to achieve the best results in adhesion.[1-3]

Currently, there is increasing interest in, and demand for, the use of all-ceramic materials due to their nonmetallic, biocompatible, and improved esthetic features. All-ceramic restorations have excellent esthetic outcomes compared with other restorative materials. Ceramic restorations are used as inlays, onlays, veneers, and crowns.[4,5]

The materials used in all-ceramic restorations include silica-based glass ceramics (feldspathic porcelain, leucite-reinforced ceramic, and lithium disilicate ceramic) and silica-free high-strength ceramics such as zirconia and alumina. Among the all-ceramic materials, zirconia and lithium disilicate are becoming the most popular materials due to improved mechanical strength. Both silica-based and silica-free ceramics have specific properties and specific directions for use, in addition to a recommended adhesive agent to achieve a strong and long-term bonding success.[4,6,7]

The clinical success of ceramic restorations is directly dependent on achieving a reliable bond strength between the cement and ceramic surfaces.[8] The practice of using silane coupling agents to enhance the bond of resin composite to silica-based ceramic is a well-accepted practice in dental technology. When silane is applied to the surface of a ceramic and then dried, an interphase layer of silane is created.[9] Silane-coupling agents are very effective in promoting adhesion for silica-based materials, such as lithium disilicate, and are used for adhesion promotion in ceramic cementation and repair with resin composites. This silane-containing primer has a hydroxyl silicon-methyl group that binds to the hydroxyl group of the silicate. It helps to form a durable bond between resin composite and silica-based ceramics.[10,11]

The aim of dental adhesives used in indirect restoration luting is to provide retention to resin cements. This retention withstands mechanical forces and prevents leakage along the margins of the restoration.[12]

Recent activity in the field of adhesive dentistry has resulted in the development of single-step adhesives that are compatible with tooth structure and different restorative materials. The single-step adhesives simplify the clinical procedures and help in avoiding bonding technique errors. The use of these adhesives offer a cost savings and help the dentist have proper control of the adhesive procedure. In the field of dentistry, they are popularly known as universal adhesives.[8,13,14]

Reports have shown that, when evaluations were done on different restorative materials used in the field of adhesive dentistry, the bonding ability of the universal adhesives is superior in comparison with other contemporary dental bonding agents.[13-16]

Many studies have reported high bond strengths when using a silane to treat the lithium disilicate before applying the bonding agent. However, only two studies have been published that compare the bond strength when using the universal adhesives alone.[8,13,14,17-19]

To provide scientific evidence for the capability of universal adhesives to bond to lithium disilicate without using a separate silane application, bond strengths need to be evaluated, including after-aging stimulation that represents the extreme conditions in the oral environment.

In this study, three commercially available universal adhesive bonding agents were selected for use in this study: Scotchbond Universal Adhesive (3M ESPE), All-Bond Universal (BISCO), and Futurabond U (Voco). The materials were all used without a separate silane to bond to lithium disilicate ceramic.

OBJECTIVE

The purpose of this study was to evaluate and compare the 24-hour and aged shear bond strength of three universal adhesives to silinated and unsilinated lithium disilicate ceramic restorative material.

HYPOTHESES

Null Hypothesis

The shear bond strength of universal adhesives to unsilinated lithium disilicate is statistically not different from silinated bond strengths at any time point.

Alternative Hypothesis

The shear bond strength of universal adhesives to unsilinated lithium disilicate is statistically less than silinated bond strengths at any time points.

REVIEW OF LITERATURE

HISTORICAL BACKGROUND

The history of chemically adhesive material dates back to 1955 as reported by Michael Buonocore[1] on the benefits of acid-etching. He was able to demonstrate that the treatment of enamel with phosphoric acid induced a porous surface that was infiltrated by resin and produced a strong micromechanical bond. However, the clinical application of acid etching was realized when resin composites became commercially available as a result of the research by Bowen's group.[1] With advanced technologies, dental adhesives have evolved from no-etch to total-etch (fourth- and fifth-generation) and, finally, to self-etch (sixth-, seventh-, and eighth-generation) systems.[20]

There are several decisive factors that influence adhesive bonding in dentistry. The adhesive interface can be influenced by the properties of the substrate, the chemistry of the adhesive, the humidity, and the operator's skill. Dental adhesive systems are commonly characterized by three stages of application of three different substances known as etching, priming, and bonding. One of the most recent developments in adhesive dentistry is the introduction of "universal" or "multi-mode" adhesives. These materials are simplified adhesives that usually contain all bonding components in a single bottle. Universal adhesives may be applied either in etch-and-rinse or in self-etching bonding approaches.[2,20]

In 2012 a new dental universal adhesive started emerging in the market. The term "universal adhesive" had different implications such as (i) it can be used in total-etch, self-etch, and selective etch techniques; (ii) it can be used with light-cure, self-cure, and dual-cure materials without a separate activator; (iii) it can be used for both direct and indirect substrates; and (iv) it can bond to all dental substrates such as enamel, dentin, composite, metal, and different types of ceramics.

In November 2011, 3M ESPE released a new Scotchbond Universal. This Scotchbond Universal requires a separate self-cure activator or a special amine-free dual-cure cement when used with dual-cure or self-cure materials, unless used with specific cements recommended by the manufacturer, such as Rely-X Ultimate. In addition, Scotchbond Universal contains silane, which will enable it to bond effectively to silica-based ceramics.

In March 2012 a Bisco scientist, Dr. Liang Chen, and his coworkers[4] developed a new All-Bond Universal that can be used in total-etch, self-etch and selective-etch techniques. It can be used also with any light-cure materials without the need of a separate activator. Furthermore, it can be used for both direct and indirect substrates with the ability to bond with any dental substrates. However, with self-cure materials, additional silane or ceramic primer is required when used to bond glass ceramics or cured composite materials as a separate, additional step.

There are many companies that produce universal adhesives, like Voco with their Futura U bonding agent. In fact, the term “universal” adhesive is not a new term; many previous bonding agents were named as “universal” adhesives, such as XP Bond-Universal Total-etch Adhesive (Dentsply) and One-Step-Universal Dental Adhesive (Bisco).[2,13,21,22]

Ceramics and Lithium Disilicate

The American Ceramic Society has defined ceramics as inorganic, nonmetallic materials, which are typically crystalline in nature. They are compounds formed between metallic and nonmetallic elements, such as aluminum and oxygen (alumina-Al_2O_3), calcium and oxygen (calcia - CaO), and silicon and nitrogen (nitride- Si_3N_4). Therefore, in dental science, ceramics are referred to as nonmetallic, inorganic structures primarily containing compounds of oxygen with one or more metallic or semi-metallic elements. These are usually sodium, potassium, calcium, magnesium, aluminum, silicon, phosphorus, zirconium and titanium.[23]

As we look into the dental history, a French dentist, De Chemant, patented the first porcelain tooth material in 1789. In 1808 Fonzi,[23] an Italian dentist, invented a "terrometallic" porcelain tooth that was held in place by a platinum pin or frame. Ash developed an improved version of the platinum tooth in 1837. Dr. Charles Land patented the first ceramic crowns in 1903.[23] Vita Zahnfabrik introduced the first commercial porcelain in 1963.[23,24]

The introduction of porcelain veneers and inlays, together with improvements in resin bonding agents, have enabled practitioners to adopt a much more conservative approach to tooth restoration. It is no exaggeration to state that the last century saw a revolution in dental esthetics. In the 21st century, the challenge of producing high-strength ceramics without sacrificing translucency may be solved. Structurally, dental silica-based ceramics contain a crystal phase and a glass phase based on the silica structure, characterized by a silica tetrahedral, containing central Si_4+ ion with four O- ions. It is not closely packed and has both covalent and ionic characteristics. The usual dental ceramic is glassy in nature with short-range crystallinity. However, true crystalline ceramics used at present in restorative dentistry are alumina and zirconia, which are among the hardest and strongest oxides known.[7,25]

Lithium disilicate is a highly esthetic, high-strength material that can be conventionally cemented and adhesively bonded. It is a unique solution to providing full contour restorations. Lithium disilicate is one of the most widely used types of glass ceramics and is highly resistant to thermal shock due to its low thermal expansion. This type of resistant glass ceramic can be processed using either a lost-wax hot-pressing technique or by CAD/CAM milling. The pressable lithium disilicate (IPS e.max Press [Ivoclar Vivadent]) is produced through a bulk-casting production process in order to create the ingots. Machineable lithium disilicate blocks are also manufactured through a similar process, but only an "intermediate" crystallization (IPS e.max CAD Ivoclar Vivadent) is attained to ensure that the blocks can be milled efficiently in a crystalline intermediate phase.[25,26]

Bonding to Lithium Disilicate

Ceramic materials are the most biocompatible materials developed for dental restorations. The combination in the early 1980s of enamel etching with phosphoric acid and ceramic etching with hydrofluoric acid initiated the development of resin-bonded ceramic restorations that provided real opportunities for achieving excellent esthetics. However, these restorations have limitations. Signs of failure include de-bonding and fracture of the material, particularly related to cementation procedures.[11,27]

Success with resin-bonded all-ceramic restorations is dependent on obtaining a reliable bond that integrates all parts of the system into one coherent structure. The preferred manner of conditioning is fitting the surface of the ceramic restoration by etching with hydrofluoric acid, followed by the application of a silane coupling agent to achieve high bond strength.[11]

Since the 1940s, silane coupling agents have been used in industry to improve bonding between organic adhesives, ceramics, and metals. However, it was not until 1977, when Eames et al.[28] suggested the use of a silane coupling agent for dental applications. The most commonly used silane in dentistry is 3-trimethoxysilylpropylmethacrylate (MPS) diluted in a water-ethanol solution. It is marketed in a pre-hydrolyzed form (one bottle) or in a form where hydrolysis can occur by mixing silane and acid (two bottles). Both types of silane coupling agent were found to perform well, even though atmospheric moisture is unfavorable to the pre-hydrolyzed silane. Silane activates a condensation reaction that leads to polymerized siloxanes, producing oligomers, which gives the solution a milky and opaque appearance.[29,30]

Testing of Bonding Strength

In restorative dentistry, the largest area of dental substrate exposed after preparation is commonly dentin. Therefore, the amount of bond strength on dentin is important for the new restoration. The effectiveness of an adhesive system to bond to dentin is generally tested with a bond strength test. The first article on bond strength tests for dental materials was published in 1965 by Bowen.[4] Since then, many more articles have been published.[8,31-34] Those articles suggested a number of experimental testing methods, such as the tensile, shear, microtensile, microshear, and push-out, and so forth.

In 2010 Scherrer et al.[34] published data about laboratory studies on six dentin adhesive systems and four laboratory methods: macroshear, microshear, macrotensile, and microtensile bond strength tests. The review revealed a large variability for the same adhesive system evaluated with the same bond strength method, not only as inter-institute variability but also as intra-institute variability. The variability was similar for each test method.[31,34]

The International Standards Organization's (ISO) Technical Specification No. 11405 provides guidance on substrate selection, storage, and handling as well as essential characteristics of different test methods for quality testing of the adhesive bond between restorative materials and tooth structure. It also presents some specific test methods for bond strength measurements. ISO 29022:2013 specifies a shear test method used to determine the adhesive bond strength between direct dental restorative materials and tooth structure (e.g., dentin or enamel). The method described was principally intended for dental adhesives. The method includes substrate selection, storage and handling of tooth structure, and the procedure for testing.

MATERIALS AND METHODS

MATERIALS SELECTION

The materials investigated in this study were three universal adhesive bonding agents selected from commercially available adhesives that use no separate silane material to bond to lithium disilicate ceramic materials: Scotchbond Universal Adhesive (3M ESPE), All-Bond Universal (BISCO), and Futurabond U (Voco). These adhesives were used to bond composite resin (Tetric ceram shade A3. Ivoclar Vivadent) to selected lithium disilicate material (e.max CAD, Ivoclar Vivadent, Amherst, NY).

The specimens were divided randomly into six groups. Each group was subdivided into four equal subgroups (n = 17), as shown in Table I. The first three groups used the universal adhesive directly. The remaining three groups used the ceramic restorative material treated with silane (Ultradent). Silane was applied and left to evaporate for 60 seconds before the universal adhesive was applied.

Sample Preparation

Blocks of lithium disilicate (e.max CAD, Ivoclar Vivadent, Amherst, NY) in bisque (blue, metasilicate) form were sectioned into rectangular coupons using a low-speed cutting diamond blade (0.4-mm thickness) (Isomet, Buehler Ltd, Lake Bluff, IL), as shown in Figure 1.

Four hundred eight IPS e.max CAD (Ivoclar Vivadent) ceramic coupons (10 x 10 x 2 mm^3) were processed and fired according to the manufacturer’s instruction, as shown in Table II. Acrylic cubes (15 x 15 mmx 20 mm^3) were placed around each ceramic coupon. The samples were mounted in the cubes by using Fastray (Bosworth Co., Skokie, IL) self-curing acrylic resin.

To establish a uniform surface, each specimen was finished and polished with a wheel rotational polishing machine using 180- and 400-grit, respectively, by silica carbide abrasive paper under a steady stream of water. All specimens were subjected to ultrasonic cleaning in distilled water for 10 minutes.

The coupons were treated with IPS Ceramic Etching Gel 5.0-percent hydrofluoric acid (Ivoclar Vivadent) for 20 seconds.

Sample Adhesive Application

The universal adhesive was applied according to manufacturer's directions, as shown in Table III and Table IV. After adhesive application, a composite resin (Tetric ceram shade A3. Ivoclar Vivadent), was placed on top of the adhesive using a bonding jig (Ultradent) to create cylinders of 2.38 mm in diameter and approximately 2 mm in height and light-cured using an Optilux 400 light cure unit (Demetron Research Corp, Danbury, CT).

Sample Testing

The first subdivided group was stored in water for 24 hours then debonded using shear force by a universal testing machine (MTS) (Figure 2) using an Ultradent notched, semicircular-shaped edge at a crosshead speed of 1.0 mm/min. The second subdivided group of specimens was exposed to water storage for one month and exposed to thermocycling 5000 cycles 5-55°C/30s dwell time (Figure 3) before being subjected to debonding. The third subdivided group was exposed to water storage for two months while exposed to thermocycling 5000 cycles 5 ºC/30s to 55ºC/30s dwell time before being subjected to debonding. The last subdivided group was exposed to water storage for three months while exposed to thermocycling 5000 cycles 5ºC/30s to 55ºC/30s dwell time before being subjected to debonding.

The shear bond strength was obtained from a computer software program, Test-Works 4.0 (MTS Systems Corporation, St. Paul, MN). Shear bond strength values in MPa were calculated by dividing the peak load by the bonded area. The values were recorded for statistical analysis.

Failure Mode Examination

Debonded specimens were examined under a stereomicroscope at X45 magnification to evaluate the fracture pattern, and the failure mode was classified as follows:

Adhesive failure at the restorative material interface.

Cohesive failure within the lithium disilicate restorative material surface.

Mixed failure - partially adhesive and partially cohesive.

In addition, to obtain qualitative information on the ceramic surface after debonding, scanning electron microscopy (SEM) (JEOL JSM-5310LV, Jeol Ltd, Tokyo, Japan) was used to examine the ceramic side of randomly selected representative specimens after a gold sputter application of each group.

Statistical Analysis

Shear bond strength results (mean, standard deviation, standard error, range) and fracture pattern (adhesive, mixed, cohesive percentages) were summarized by group and time. The effects of the adhesive bonding agent to the ceramic restorative materials on shear bond strength were evaluated using 3-way ANOVA, with factors for type of universal adhesive material, presence or absence of silane, and time, as well as all two-way and three-way interactions among the factors. Pair-wise comparisons were made using Fisher's Protected Least Significant Differences to control the overall significance level at 5 percent. The estimated within-group standard deviation was 5 MPa. With a sample size of 17 per group for each storage time, the study had 80-percent power to detect a difference of 5 MPa between any two groups, assuming two-sided tests each conducted at a 5-percent significance level.

RESULTS

SHEAR BOND STRENGTH

The three-way interaction between adhesive, silane, and time was statistically significant ($p < 0.0001$), indicating comparisons of one factor will depend on the levels of the other factors, as shown in Table V. The results showed that the shear bond strength was higher with silane than without silane ($p < 0.0001$), regardless of the levels of adhesive or time (Figure 4).

All-Bond had significantly higher shear bond strength than Scotchbond ($p < 0.0001$) for all silane-time combinations except 24 h with no silane ($p = 0.83$). Without silane, All-Bond had significantly higher shear bond strength than Futurabond ($p < 0.0001$); however, with silane, All-Bond had significantly higher shear bond strength than Futurabond at 24 h ($p < 0.0001$) and was not different at 1 m ($p = 0.22$) or 2 m ($p = 0.08$), but was significantly lower than Futurabond at 3 m ($p = 0.0043$). Futurabond had significantly lower shear bond strength than Scotchbond at 24 h ($p = 0.0001$), significantly higher shear bond strength at 1 m ($p < 0.0001$) and 3 m ($p < 0.0001$), and significantly higher shear bond strength at 2 m with silane ($p < 0.0001$) but not without silane ($p = 0.89$), as shown in Figure 5, Figure 6, and Figure 7.

Shear bond strength was significantly higher at 24 h than 1 m for All-Bond ($p < 0.0001$) and Scotchbond ($p < 0.0001$), but for Futurabond 24 m was higher than 1 m only without silane ($p = 0.0194$). Shear bond strength was significantly higher at 24 h and 1 m than at 2 m ($p < 0.0001$) or 3 m ($p < 0.0001$) regardless of the adhesive or the presence of silane. Shear bond strength was significantly higher at 2 m than 3 m for All-Bond ($p < 0.0001$) and Scotchbond ($p = 0.0330$), but for Futurabond 2 m was higher than 3 m with silane (p = 0.0232) but not without silane (p = 0.68), as shown in Figure 8 and Figure 9.

Failure Mode

For all the examined debonded specimens, the fracture pattern and the failure mode was mixed failure – partially adhesive and partially cohesive.

SEM Results

Figure 10 shows the SEM result and image descriptions of the ceramic side for randomly selected failed samples. The SEM result shows agreement with the stereomicroscope result regarding the mixed failure mode.

FIGURES AND TABLES

FIGURE 1. Isomet 1000, Buehler, Lake Bluff, USA.

FIGURE 2. Universal testing machine (MTS Sintech Renew 1123, Eden Prairie, MN).

FIGURE 3. Thermocycler 1100, Miesbacher St 34, Germany.

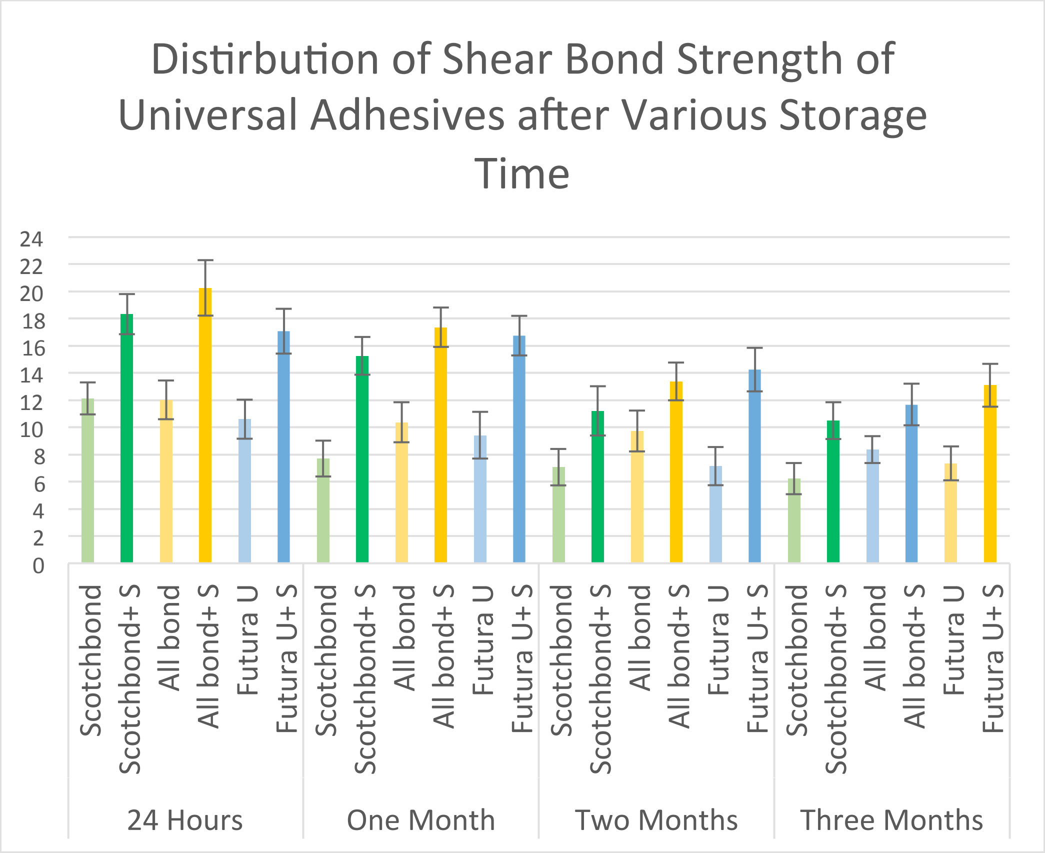

FIGURE 4. Distribution of shear bond strength of universal adhesive after various storage times.

FIGURE 5. Comparison of shear bond strength of Scotchbond Universal Adhesive after various storage times.

FIGURE 6. Comparison of shear bond strength of All-Bond Universal Adhesive after various storage times.

FIGURE 7. Comparison of shear bond strength of Futura U Universal Adhesive after various storage times.

FIGURE 8. Comparison of shear bond strength between different universal adhesives applied without silane after various storage times.

FIGURE 9. Comparison of shear bond strength between different universal adhesives applied with silane after various storage times.

FIGURE 10. SEM image of apparently mixed failure under light microscopy;
SEM images show a mixed type of failure where both composite filler (A) and bonding agent (B) can be identified on the fractured ceramic surface (C).

TABLE I

Group description used in the study

Group number	Material	Time
1	Scotchbond universal	24 hours
2	Scotchbond universal	1 month
3	Scotchbond universal	2 months
4	Scotchbond universal	3 months
5	Silane + Scotchbond universal	24 hours
6	Silane + Scotchbond universal	1 month
7	Silane + Scotchbond universal	2 months
8	Silane + Scotchbond universal	3 months
9	All-bond universal	24 hours
10	All-bond universal	1 month
11	All-bond universal	2 months
12	All-bond universal	3 months
13	Silane +All-bond universal	24 hours
14	Silane +All-bond universal	1 month
15	Silane +All-bond universal	2 months
16	Silane +All-bond universal	3 months
17	Futurabond U	24 hours
18	Futurabond U	1 month
19	Futurabond U	2 months
20	Futurabond U	3 months

(continued)

TABLE I (cont.)

Group description used in the study

Group number	Material	Time
21	Silane + Futurabond U	24 hours
22	Silane + Futurabond U	1 month
23	Silane + Futurabond U	2 months
24	Silane + Futurabond U	3 months

TABLE II

E.max CAD processing instruction

Program at CS Program	Furnace
403/757	Stand-by temperature [°C/°F]
6:00	Closing time [min]
90/162	Heating rate [°C/°F/min]
820/1508	Firing temperature T1 [°C/°F]
0:10	Holding time H1 [min]
30/54	Heating rate [°C/°F/min]
840/1544	Firing temperature T2 [°C/°F]
7:00	Holding time H2 [min]
550/820 1022/1508	Vacuum 1 11 [°C/°F] 12 [°C/°F]
820/840 1508/1544	Vacuum 2 21 [°C/°F] 22 [°C/°F]
700/1292	Long-term cooling L [°C/°F]

TABLE III

Materials used in the study

Name	Manufacturer	Batch	Composition
Scotchbond Universal Adhesive	3M ESPE	41254	Bisphenol A glycidyl methacrylate Hydroxyethyl Methacrylate Decamethylene Dimethacrylate Ethanol Water Silane treated Silica Propenoic Acid, Methyl-Decanediol and Phosphorous OXIDE (P_2O_5) Copolymer of Acrylic and Itaconic acid Dimethylaminobenzoat Camphorquinone (Dimethylamino)Ethyl methacrylate methyl ethyl ketone
All-Bond Universal	Bisco	1200006111	MDP*[2], Bisphenol A glycidyl methacrylate, Hydroxyethylmethacrylate, ethanol, water, initiators
Futurabond U	Voco	1572	Hydroxyethylmethacrylate, Bisphenol A glycidyl methacrylate, HydroxyethyldiMethacrylate, Urethanedimethacrylate, Acidic adhesive monomer, Ethanol, initiator, catalyst
IPS e.max Cad	Ivoclar Vivadent	605330	Silica oxide (SiO_2) Additional contents: Lithium oxide (Li_2O), Potassium oxide (K_2O), Magnesium oxide (MgO), Aluminum oxide (Al_2O_3), Phosphorus oxide (P_2O_5) and other oxides.
IPS ceramic etching gel	Ivoclar Vivadent	531548	Hydrofluoric Acid
Silane coupling agent	Ultradent	10324	Methacryloxy propyl trimethoxy silane Isopropyl Alcohol

TABLE IV

Directions of use for universal adhesive bonding agents used in study

Name	Direction
Scotchbond Universal Adhesive	Rinse the surface with water and dry with water-free and oil-free air or with cotton pellets. In combination with other composite cements: - Place one drop each of Scotchbond Universal and Scotchbond Universal DCA in a mixing well and mix for 5 sec. - Immediately after mixing, use the disposable applicator to apply the adhesive to the entire surface of the restoration to be cemented and allow it to react for 20 sec. Do not light-cure. - Follow the instructions for use from the manufacturer to apply the cement.
All-Bond Universal	Apply 1 coat of ALL-BOND UNIVERSAL and air dry to remove excess solvent. Light cure for 10 seconds.
Futurabond U	Clean thoroughly with water spray and dry with moisture- and oil-free air. Activating Futurabond U SingleDose: Detach a SingleDose blister at the perforation and turn the printed side up. Hold the SingleDose blister between thumb and forefinger and, by pressing on the area marked “press here”, allow the liquid contained in the blister to flow into the mixing and dispensing chamber. Position the enclosed Single Tim applicator in the center of the colored circle in order to pierce through the film of the mixing and dispensing chamber. Expand the opening to its maximum size using a circular motion. By stirring thoroughly with the applicator, create a homogeneous, streak-free mixture of the two liquids. Apply the adhesive homogeneously to the surface and rub in for 20 s using the applicator. Dry off the adhesive layer with dry, oil-free air for at least 5 s in order to remove any solvents. Cure the adhesive layer for 10 s using a commercially available polymerization device

TABLE V

Statistical summary of shear bond strength

Adhesive	Silane	24h Mean MPa (SE)[SD]	1m Mean MPa (SE)[SD]	2m Mean MPa (SE)[SD]	3m Mean MPa (SE)[SD]
All-Bond	N2*	12.0 (0.3)[1.4]A[$]a	10.4 (0.4)[1.5]Ab[#]	9.7 (0.4)[1.5]Ac	8.4 (0.2)[1.0]Ad
	Y1	20.3 (0.5)[2.0]Aa	17.4 (0.3)[1.4]Ab	13.4 (0.3)[1.4]Ac	11.7 (0.4)[1.6]Bd
Futura U	N2	10.6 (0.4)[1.5]Ba	9.4 (0.4)[1.7]Bb	7.1 (0.4)[1.4]Bc	7.4 (0.3)[1.3]Bc
	Y1	17.1 (0.4)[1.6]Ca	16.7 (0.4)[1.4]Aa	14.3 (0.4)[1.6]Ab	13.1 (0.4)[1.6]Ac
Scotchbond	N2	12.1 (0.3)[1.2]Aa	7.7 (0.3)[1.3]Cb	7.1 (0.3)[1.3]Bc	6.2 (0.3)[1.1]Cd
	Y1	18.3 (0.4)[1.5]Ba	15.3 (0.3)[1.4]Bb	11.2 (0.4)[1.8]Bc	10.5 (0.3)[1.4]Cd

* Different numbers represent statistical significant difference within each type of bonding agent used at all-time points based on three-way ANOVA.

[$] Different upper case letters indicate statistical significant difference between different types of bonding within each silane condition at each time point based on three-way ANOVA.

[#] Different lower case letters indicate statistical significant difference between different time points within each type of bonding agent used and silane condition based on three-way ANOVA.

DISCUSSION

Leucite-reinforced glass ceramics (LRGC), developed in the early 1990s, employed leucite crystals in an amorphous glass matrix (IPS Empress 1, Ivoclar Vivadent). The leucite particles increased the strength of the material through the limitation of crack propagation. The ability to acid-etch the surface and adhesively bond to tooth structure greatly improved the predictability and survival of these restorations, and it revolutionized modern-day esthetic dentistry. A new generation of monolithic lithium-disilicate glass-ceramic material (IPS e.max CAD, Ivoclar Vivadent) was developed with flexural strength values between 360 MPa and 400 MPa for CAD and pressed restorations, respectively.[35,36]

The three-way interaction between adhesive, silane, and time was significant, indicating that some comparisons of one factor depend on the levels of the other factors. Shear bond strength was greater with silane than without silane regardless of the levels of adhesive or time. Therefore, we can reject the null hypothesis that the shear bond strengths of universal adhesives to unsilinated lithium disilicate is not statistically significant to silinated bond strengths at any time point.

The results of this study revealed that the silinated Scotchbond Universal had significantly higher shear bond strength than unsilinated Scotchbond Universal for almost all storage times. In our study, the result of Scotchbond improved from 12.1 MPa to 18.3 MPa. This is a similar percentage, although there is a difference in values probably related to differences in the method of application to the study by Panah et al.,[19] which showed the shear bond strength improved from 14.04 MPa to 24.70 MPa when silane was applied and that the samples were stored in distilled water for 24 h. Additionally, a study by Kalavacharla et al.[14] showed the improvement from 19.08 MPa to 40.47 MPa with exposure to 10,000 thermocycles compared with this study, which showed with exposure to 5000 thermocycles improvements with silane application from 7.7 MPa to 15.3 MPa, 7.2 MPa to 11.2 MPa, and 6.2 MPa to 10.5 MPa for one, two, and three months respectively. This study further confirmed that lithium disilicate should undergo silinization prior to bonding.[14,19]

The main difference in the composition of these universal adhesives is the incorporation of silane, which is only present in Scotchbond. Therefore, Scotchbond was expected to produce greater bond strength than All-Bond and Futura U; however, the Shear bond strength values obtained for Scotchbond without silane application were the same or less than All-bond and Futura U (Table V). This implies that the silane contained in Scotchbond failed to produce any significant chemical bonds with the ceramic. This finding corroborates the results of Kalavacharla et al.,[14] who compared the effect of Scotchbond with and without silane application on lithium-disilicate bond strength. They reported that the bond strength was significantly improved when silane was applied prior to the application of the universal adhesive; thus, the incorporation of silane in the universal adhesive itself would seem ineffective in improving the ceramic-resin bond. This could be explained by the presence of a mixture of various components within the same bottle, as it has been reported that bis-GMA may inhibit the action of silane by disrupting the condensation reaction with the hydroxyl group of a silica-based ceramic.[37,38] Furthermore, the acidic functional monomer 10-MDP (methacryloyloxydecyl dihydrogen phosphate) that is incorporated in universal adhesives may impede the ideal chemical interaction between silane and ceramics owing to the tendency for premature hydrolysis in an acidic environment.[39]

Although clinical trials produce the most reliable evidence, *in-vitro* adhesion tests provide immediate information on the bonding effectiveness of new materials. The most commonly employed bond strength tests are tensile and shear tests.[40,41] However, conventional shear and tensile tests have been criticized for using relatively large bonded surfaces. In this regard, the microtensile technique is considered more reliable, being able to more closely reflect the interfacial bond strength, as it offers more uniform stress distribution.[41,42] Since its introduction by Sano et al. in 1994, the microtensile method has undergone several improvements and various applications for *in-vitro* bond strength testing. However, a possible limitation of this technique emerged when testing the bond strength to tooth structure or restorative materials. A high frequency of premature failures and large standard deviation values were reported.[41,43,44]

Recently, the microshear bond strength test was introduced as an alternative to the conventional shear test.[40] Similar to the microtensile test, the microshear technique involves testing of small areas, and allows preparation of multiple specimens from the same material. However, sectioning and trimming steps, which may introduce early microcracking within the specimen, are avoided.[41,45] Ishikawa et al compared the microtensile and microshear methods for testing adhesion to enamel and dentin of all-in-one adhesives. Those authors' conclusion was that microshear was more effective at detecting differences in the bonding conditions produced by adhesives of this category.[41,46]

Guarda et al. conclude in their article to that water storage with thermocycling caused deterioration in bond strength of the tested bonding strengths regardless of the adhesive or the presence of silane.[36] This study clearly showed that the shear bond strength was significantly higher at 24 h and 1 m than at 2 m or 3 m. Shear bond strength was significantly higher at 2 m than 3 m for All-Bond and Scotchbond, but for Futurabond U 2 m, it was higher than 3 m with silane but almost equal without silane.

The quality of the bond should not be evaluated based on the bond strength data only. The mode of failure analysis provides the necessary information for adhesive abilities and limitations, which is the important test of any bonding system. Moreover, SEM micrographs of fractured surfaces revealed that all fractures that occurred were considered as mixed failures regardless of silane applications or not. The result of our study was in accordance with Kalavacharla et al. showed that, when the silane is applied, the mode of failure was considered as mixed failure. On the other hand, the contents of the universal adhesives may be enough to obtain considerable bond strength that leads to making the fractures involve the three layers of the bonding system.

One limitation of this study was that the thermocycling and water storage only represent months after application. And in the clinical scenarios, they may fail after years. Further investigation and study need to be considered with a different method of testing, such as microtensile bond strength. However, the universal adhesive shows a promising future for simplifying the clinical technique for providing a durable bond between all restorative materials and tooth structures. The use of silane prior the universal adhesive bonding application to lithium disilicate restorative material is necessary to provide an excellent bond strength.

SUMMARY AND CONCLUSION

Within the limitations of this study, it was concluded that:

The optimal bonds to lithium disilicate are achieved by application of silane prior to application of a universal adhesive. Although the constituent silane in the universal adhesive was not effective in optimizing the resin to ceramic bond, silane should always be applied to lithium disilicate prior to bonding.

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ABSTRACT

EFFECTIVENESS OF UNIVERSAL ADHESIVE BONDING AGENTS ON THE SHEAR BOND STRENGTH TO LITHIUM DISILICATE CERAMICS

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Background: All-ceramic restorations have excellent esthetic outcomes compared with other restorative materials. Lithium disilicate is classified as one of many silica-based all-ceramic materials. Currently, companies have provided single-step adhesives, known as universal adhesives, compatible with different restorative materials including lithium disilicate. Many studies have reported greater bond strengths when using a silane to treat the lithium disilicate before applying the bonding agent. Moreover, few studies were published comparing the bond strength when using the universal adhesive alone.

Purpose: The objective of this study was to evaluate and compare shear bond strength of three universal adhesives to lithium disilicate ceramic restorative material.

Materials and Methods: Three universal adhesive bonding agents were selected from commercially available adhesives. 408 IPS e.max CAD ceramic discs were processed, fired, and etched for 20s. The specimens were divided into six groups. The first three groups used the universal adhesive directly. The remaining three groups were treated with silane. Then, a composite resin cylinder was placed on top of the adhesive using a bonding jig. Each group was subdivided into four equal subgroups ($n = 17$), subjected to different aging simulation procedures: 24 h, one month with 5000 thermocycles, two months with 5000 cycles, and three months with 5000 cycles. Then, specimens were debonded using shear force by a universal testing machine (MTS).

Results: Shear bond strength was greater with silane than without silane ($p < 0.0001$), regardless of the levels of adhesive or time. Shear bond strength was significantly greater at 24h and 1m than at 2m ($p < 0.0001$) or 3m ($p < 0.0001$) regardless of the adhesive or the presence of silane. Debonded specimens were examined under a stereomicroscope at X45 magnification to evaluate the fracture pattern. SEM was used to prove the results were considered as mixed failure.

Conclusion: The optimal bonds to lithium disilicate are achieved by application of silane prior to application of a universal adhesive. Although the constituent silane in the universal adhesive was not effective in optimizing the resin

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