Evaluation of the effect of surface treatments on shear bond strength between lithium disilicate ceramic and dentin.

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ABSTRACT

Purpose: To evaluate the effect of different surface treatments on shear bond strength between dentin and IPS e.max lithium disilicate glass-ceramic.

Materials and Methods: Eighteen extracted third molars were embeded in epoxy resin. The tooth was sectioned vertically in mesiodistal direction using a low speed hard tissue microtome. The buccal and lingual surfaces of each section were ground flat using 600 grit Silicone carbide paper. Eighteen ceramic discs consisted of lithium disilicate glass-ceramic were prepared with a diameter of 4.7mm and height of 2.2mm. The discs were divided in two groups (n=10): (1) IPS e.max treated with hydrofluoric acid and Monobond Plus (MBP) and (2) IPS e.max treated with Monobond Etch &Prime (MBEP). The tooth was cemented with Multilink Automix and stored for 24hours at room temperature before thermocycling and subsequently loaded to failure in Universal Testing Machine. Failure mode were recorded for each specimen.

Result: Bond strength analysis and t-test analysis MBEP demonstrated the higher shear bond strength (SBS). MBP and MBEP showed no statistically significant difference were found between them. One-way ANOVA and t-test was used to determine differences in bond strength within and between the groups. Cohesive failure in resin cement was predominant with higher results while adhesive and mixed with lower and equal.

Conclusion: Surface treatment with Monobond Etch and Prime has a favorable effect on SBS between dentin and lithium disilicate glass-ceramic with resin cement compared with Monobond Plus.

Keywords: Lithium disilicate glass-ceramic, Shear bond strength, Monobond Plus. **(J Bagh Coll Dentistry 2017; 29(3):1-8)**

INTRODUCTION

The increasing esthetic demands of conservative dentistry led to the launch of new materials and techniques. A major development in the field was the introduction of lithium disilicate ceramics a group of etchable glass-ceramics stronger than feldspathic porcelain, with exceptional esthetics that can establish a strong micromechanical bond with methacrylate based resin luting agents. In addition, by using methacrylate functionalized silane primers on the etched surface, chemical bonding is mediated with the methacrylate resin matrix of the luting agents [1,2].

Silanization transforms the hydrophilic etched-ceramic surface to hydrophobic, promoting thus the wettability of the hydrophobic resin luting agent on the silane treated surface and improves bond strength in comparison with etched, but not silanated surfaces [3].

Bonded restorations have important advantages over conventionally cemented since they effectively reduce marginal defects and require cavity preparation with minimal removal of sound dental tissues [4]. The standard procedure for bonding lithium disilicate ceramics involves two separate steps of ceramic surface treatment. The first step includes chemical etching with hydrofluoric acid (HF-acid), water rinsing, acid neutralization, water rinsing again and air drying. Then follows the second step where the silane primer is applied, left intact and air-dried.

To reduce the steps required for ceramic surface preparation, a new ceramic conditioning agent was introduced in early 2015 (Monobond Etch&Prime Ivocar Vivadent AG), which integrates the etching and silane priming treatments in a single step. These materials shortened the treatment time of the clinical steps by etch and silanate glass-ceramic surfaces in one working step. Furthermore, the technique sensitivity or inaccuracy of the pre-treatment of glass-ceramic restorations compared with conventional conditioning is reduced.

Etching the inner surface of the porcelain veneer with hydrofluoric acid creates a retentive etch pattern. SEM of etched porcelain surface showed an amorphous micro-structure with numerous porosities [5,6,7,8,9].

These micro-porosities increase the surface area for bonding and lead to a micro-mechanical interlocking of the resin cement. Several factors like the etching time, concentration of the etching liquid, fabrication method of the porcelain restoration [10,11], and type of porcelain [12,13] determine the micro-morphology of the etch pattern and consequently the bond strength of the resin cement to the etched porcelain [14].

Multilink Automix is used in combination with HF-acid and Monobond Plus or can be used in

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combination with self-etching and self-curing Multilink Primer. This primer is responsible for establishing a strong adhesive bond to the tooth structure. The initiator contained in the primer permits chemically initiated polymerization (self-curing), which is accelerated when the resin comes into contact with the primer. Furthermore, the presence of a photo-initiator enables final polymerization with light. Bonding of resin to dentine is based on resin infiltration into dentine tubules and bonding the collagen fibers of the dentine to form a hybrid layer. This layer is considered essential to create a strong and reliable bond between resin and dentine [15].

Therefore, the aim of this study is evaluating the shear bond strength of all ceramic with different surface treatment bonded to dentine using Monobond Plus or Monobond Etch & Prime together with the Multilink Automix cement system and the bond failures.

The null hypothesis was that Monobond Etch & Prime would result in bond strength that is comparable with that of 5% HF acid etch and treated by Monobond Plus.

MATERIALS AND METHODS

Tooth preparation:

Extracted intact third molars, stored at 8 °C in tap water containing 0.5% sodium azide, were embed in epoxy resin, up to the cervical region. Each tooth is sectioned vertically in mesiodistal direction using a low speed hard tissue microtome (Isomet, Buhler, Evanstone, IL, USA). The cutting was performed under water coolant.

The buccal and lingual surfaces of each section were cut lat using 600 grit Silicone Carbide Paper. The cutting surfaces were covered with an adhesive tape (50µm thick), providing holes (4mm diameter) located at the center of the specimens to standardize the bonding area. Eighteen dentin specimens were prepared and randomly divided in two groups of nine specimens for each. The surfaces were thoroughly cleaned using the medium-grit paste (Proxyt RDA 36, Ivoclar Vivadent) indicated for the cleaning of cavities.

Preparation of lithium disilicate glass-ceramic discs:

Eighteen ceramic discs consisted of lithium disilicate glass-ceramic (IPS e.max Press, Ivoclar-Vivadent AG, Schaan, Liechtenstein) were prepared with a diameter of 4.7mm and height of 2.2mm according to (SS-EN ISO 6872:2015 (E)). The discs were divided in two groups (MBP, MBPE) and treated as follows:

1) MBP group

Acid etching was performed by 5% HF (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 30 seconds, residual acid were neutralized in powder for 60 seconds, then water-rinsed for 30 seconds and finally air-dried for10 seconds. Silanization was performed by applying a silane agent with phosphate and disulphate monomers (Monobond Plus, Ivoclar Vivadent) for 60 seconds followed by 10 seconds air drying. This treatment was used as the control group.

2) MBEP group

A new primer for simultaneous etching and silanization was used (Monobond Etch &Prime, Ivoclar Vivadent). The ceramic surface was primed with the new agent by agitation with a microbrush for 20 seconds, the primer was allowed to react for 40 seconds, rinsed with water for 30 seconds and finally air-dried for 10 seconds.

Bonding of discs to the dentin:

All treated discs were cemented to dentine using a resin luting agent (Multilink Automix, Ivoclar Vivadent). Dentine specimens were treated with the corresponding primers and then the ceramic disks were bonded to Dentin. To control the luting agent thickness, constant force of 15 N was applied for 1 mint. Resin excess was removed and then light-cured from four directions for 20 seconds with a curing halogen device (Heraeus Translux®PowerBlue®, Heraeus Kulzer GmbH, Hanau, Germany), emitting1000 mW/cm^2 light intensity. The curing time was set at 20 seconds for each of four directions 90° apart and finally for 60 seconds with the seating load removed and stored for 24 h at room temperature before thermocycling.

Artificial ageing – Thermocycling (TC):

All specimens were after water storage then subjected to thermal-cycling (TC) under the following conditions: 5000 cycles, 5°C/55°C, 1cycle/min, 20 seconds' dwell time, 10 seconds' transfer time. Each cycle lasted 60 seconds. The specimens were subsequently loaded to failure under shear stress applied at the interface using a knife-edge loading head at a cross-head speed of 0.5 mm/min, until the ceramic disc was dislodged from the tooth. Maximum load to failure was recorded in Newton (N) for each sample and then shear bond strength was expressed in Megapascals (MPa) by dividing the load at failure (Newtons) by the bonded surface area.

$$S = F\ max/A$$

Where: S= Shear bond strength (MPa). F= load at failure (N). A= $\pi\ r^2$.

The debonded ceramic surfaces were examined under a stereomicroscope lens (Wild M3, Wild Heerbrugg, Switzerland) at 6.4× magnification to assess the failure modes. The type of failures was classified as resin cohesive, adhesive (at the dentin-resin interface), or mixed (combination of adhesive and cohesive).

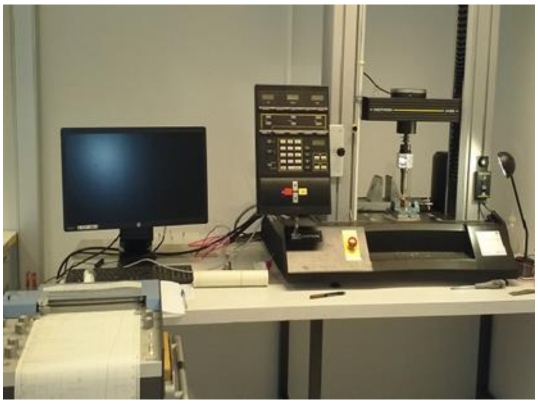

Figure (1) Universal testing machine

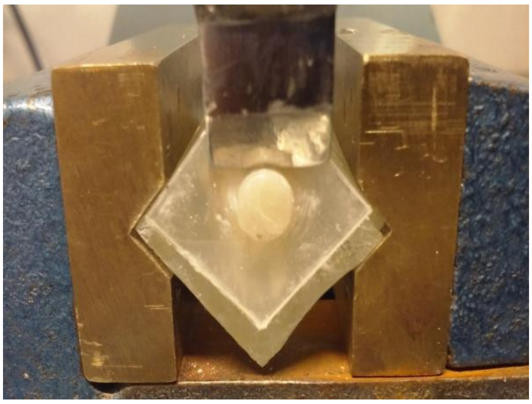

Figure 2: Sample holder.

Failure mode:

To classify the type of the failure, which could be either cohesive, adhesive, or mixed (combination of adhesive and cohesive failure).

Statistics analysis:

The SPSS software was used to perform the statistical analysis. One way analysis and normality test (Shapiro-Wilk) and was used to determined differences in bond strength within and between the groups (IBM SPSS Statistics 20.0, SPSS Inc., Chicago, IL, USA). The level of significance was set to α = 0.05.

RESULTS:

Shear bond strength:

The mean (and standard deviation) of shear bond strength are presented in Table 1. MBEP demonstrated the highest bond strength values. No statistically significant difference was found between the two different surface pretreatments (MBP and MBEP) (P = 0.551).

Table 1: Comparison of shear bond strength (MPa) between the two groups showing mean, standard deviation, and standard error of mean.

Groups	N	Missing	Mean	SD	SE
MBP	9	0	9.629	3.182	1.061
MBEP	9	0	10.482	2.739	0.913

Difference -0.853

t = -0.610 with 16 degrees of freedom. (P = 0.551)

95 percent confidence interval for difference of means: -3.820 to 2.114

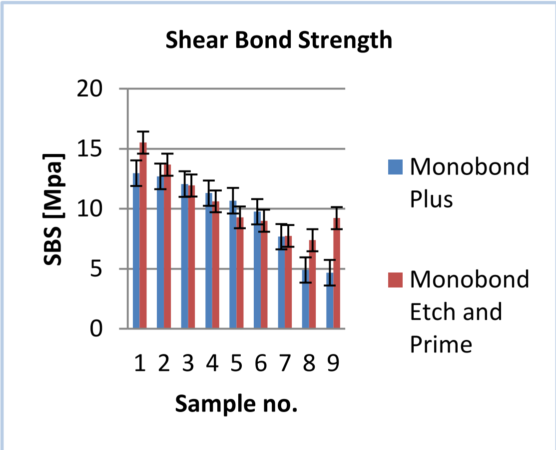

Figure 3: Mean shear bond strength values of the tested groups showing the shear bond strengths with standards error between the groups

Failure mode:

Figure 4,5 shows stereomicroscope images of the fracture surfaces of the dentin and ceramic substrates at a magnification of 6.4X. The distribution of the different failure mode of both groups MBP and MBEP revealed that the predominant mode of failure (66.67% and 55.56 %) was cohesive failure, (22.22 %) adhesive for both and the remaining (11.11% and 22.22 %) mixed failure.

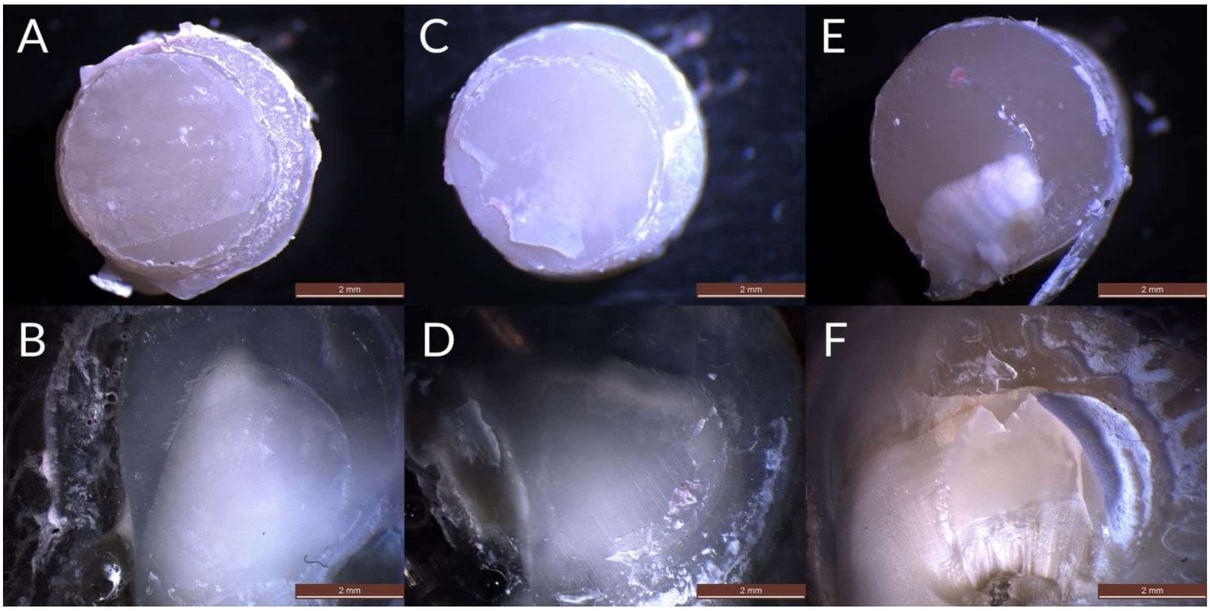

Figure 4: Representative images of failure mode of ceramic surface (CS) (ACE) and dentin surface (DS) (BDF) in group MBP (A,B) Adhesive; (C,D) Cohesive; (E,F) Mixed.

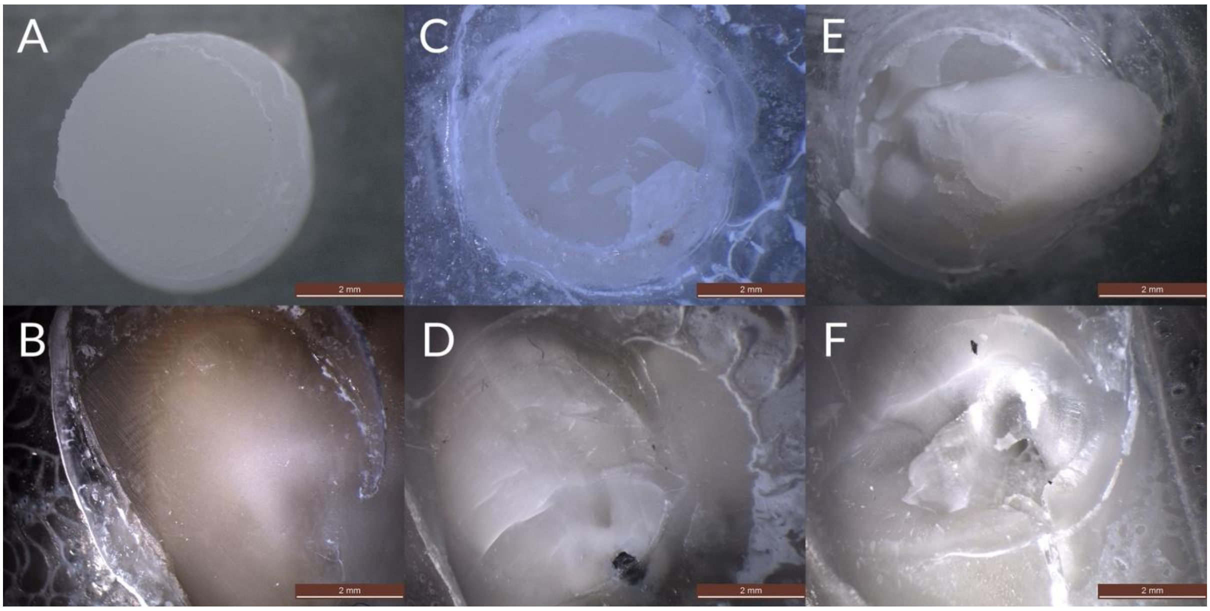

Figure 5: Representative images of failure mode of ceramic surface (CS) (ACE) and dentin surface (DS) (BDF) in group MBEP (A,B) Adhesive; (C,D) Cohesive; (E,F) Mixed.

DISCUSSION:

According to the results of this study the null hypothesis must be accepted. There are no significant differences in the shear bond strength (SBS) values between the treated groups. Although bond strength tests are not fully standardized, they are considered essential to examine factors related to bonding effectiveness as well as for the screening of new materials [16]. SBS tests are one of the most commonly used for testing dentin adhesion [17]. This popularity may be related to the simplicity of specimen preparation. However, large bonded areas more than 0.8 mm diameter of the bonded area (macroshear) may include critical flaws at the interfaces resulting in lower bond strength value as compared to the small bonded area of microshear and microtensile [16,18]. Moreover, there are concerns about the non-uniform stress generated along the interface as a consequence of specimen's geometry and loading condition.

In the present study, ceramic specimens were bonded to dentin with the luting cement to simulate the clinical situation. SBS to dentin with the hybridization mechanism ranges between 22 and 35 MPa. This strength is theoretically higher than that of enamel, because dentin is more resistant to shear fracture. The presence of more water in dentin than enamel, may affect the clinical longevity of dentin bonding. The success of all-ceramic restoration is greatly determined by the strength and durability of the formed bond between the three different components of the bonded area the tooth surface, cement and the ceramic.

The mechanism of dentin adhesion, enhanced by hybrid layer formation between the resin and dentin, was proposed by Nakabayashi et al (1982) [19]. Formation of hybrid layer is considered essential to create a strong bond between the resin and dentin. The penetration of adhesive monomers into the superficially demineralized dentin and subsequent polymerization are indispensable to create one of an ideal hybrid layer. When dentin is acid etched, the apatite phase of the smear-layer and of the underlying dentin is solubilized to permit exposure of the underlying collagen fibrils. These may leave spaces for bonding resin to penetrate [20]. The extend of resin infiltration depends on the amount of apatite removed by conditioning and the moisture of dentin. The intrinsic moisture, i.e. the outward flow of dentinal fluid, may interfere with monomer infiltration into the dentin, depending on the monomer composition of the DBS's [15].

Theoretically, the higher the tubule density and the more the tubules are widened by the etchant, the greater is the chance of obtaining a reliable bond because of the increase in the number and diameter of resin tags [21, 22]. This can be true only for bonding systems in which an acid etching step is included and is important especially for crown preparations in which approximately two million tubules are exposed [23]. There is little data published on the contribution of the tubule density to resin bonds. Gwinnett et al. [24] showed that the resin-reinforced (or hybridized) collagen network did not contribute any significant quantitative value per se to dentin bonding with an enamel–dentin bonding system. Pashley et al. [21] correlated the dentin substrates at different depths with the bond strength using a theoretical model. The resulting calculations indicated the potential for higher bond strengths to deep dentin than to superficial dentin and the importance of resin tags in the development of strong bonds. This may explain the low values of bond strength in our study as we used the superficial dentin.

By etching the inner side of the ceramic discs with hydrofluoric acid creates retentive etch pattern subsequently silanizing the etched surface. These micro-porosities increase the surface area for bonding and lead to a micro-mechanical interlocking of the resin cement. In addition to micro-porosities, micro-cracks were observed that grow when the etching time increases [25]. These cracks can act as sources of crack initiation and slightly, although not significantly, decrease the flexural strength of the etched porcelain. Weakening of the porcelain by etching was also noted in other in vitro studies [26, 27].

Silanization of etched porcelain with a bi-functional coupling agent provides a chemical link between the luting resin composite and porcelain. A silane group at one end chemically bonds to the hydrolysed silicon dioxide at the ceramic surface, and a methacrylate group at the other end with the adhesive resin. Single-component systems contain silane in alcohol or acetone and require prior acidification of the ceramic surface with hydrofluoric acid to activate the chemical reaction. With two-component silane solutions, the silane is mixed with an aqueous acid solution to hydrolyse the silane, so that it can react directly with the ceramic surface. If not used within several hours, silane will polymerise to an unreactive polysiloxane [28]. Several authors reported differences in bond strength dependent on the silane treatment used [29, 30, 31 and 32]. In addition, heating of the silane-coated porcelain to 100°C resulted in bond strength twice as high than if no heating was used [33].

Monobond Etch&Prime significantly shortens the treatment time for all ceramic materials compared with the conventional procedure. Enabling users to apply the same contact time for all materials reduces the risk of errors. The reason why Monobond Prime&Etch achieves similar bond strengths as the combination of HF etching and Monobond Plus even if it produces a less pronounced etching pattern lies in the fact that the ammonium polyfluoride ions induce the formation of reactive silanol groups. When the ceramic is rinsed, the polyfluoride is removed and the silanol groups are no longer stabilized. This gives way to a highly effective functionalization process that offsets the less pronounced etching pattern [34].

The mode of failure in this study was predominantly cohesive failure (66.67%) followed by adhesive failure at the dentin-cement interface/mode (16.66%) and mixed failure (16.66%). Correlating with findings of other researchers [35], the ceramic bond strength can be therefore interpreted to be stronger than the dentin-cement bond strength.

The present study indicated that the use of the self-etching glass-ceramic primer as a pre-treatment of ceramic enhanced the adhesion between ceramic and resin cement. This treatment is an alternative treatment to sandblast-particle-abrasion and avoids micro-crack formation and phase transitions that are detrimental to the longevity of the ceramic restoration. However, the monobond Etch& Prime treatment still requires further studies with use different types of resin composite cements. All the samples there were no adhesive mode of failure with the ceramic surface, it is still strong enough to produce sufficient microretention for a reliable adhesive bond, as confirmed by the bond strength measurements.

CONCLUSION:

The new glass-ceramic primer is a self-etching single component without using hydrofluoric acid that produces an equivalent SBS and failure mode. Monobond Etch & Prime more safe, simple in treatment and require less steps.

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الخلاصة:

الغرض من هذه الدراسة لتقييم تأثير العلاجات السطحية المختلفة بين العاج و e.max الليثيوم ديسيليكات سيراميك المزجج.

المواد والطرق تم تضمين ثمانية عشر من الأضراس المستخرجة الثالثة في راتنجات الايبوكسي. وقطعت الأسنان مقطعا عموديا في الاتجاه السطح القريب والوحشي باستخدام مشرح الأنسجة الصلبة ذو السرعة المنخفضة. تم تلميع الأسطح الشدقية واللسنية من كل قسم شقة باستخدام الرقم 600 ورقة السيليكون كربيد. تم تحضير ثمانية عشر أسطوانة من السيراميك تتكون من الليثيوم ديسيليكات سيراميك المزجج أعدت بقطر 4.7 ملم وارتفاع 2.2ملم. تم تقسيم الأقراص إلى مجموعتين الاولى (العدد=9): (e.max تعامل مع حامض الهيدروفلوريك و Monobond Plus) والمجموعة الثانية (e.max تعامل مع Monobond etch & Bond). تم لصق السيراميك مع الأسنان باستخدام Multilink Automix وتخزينها لمدة 24 ساعة في درجة حرارة الغرفة قبل وضعها في جهاز التبديل الحراري وتحميلها لاحقا إلى الفشل في آلة اختبار العالمي. تم تسجيل وضع الفشل لكل عينة.

ألنتيجة تحليل قوة الالتصاق وتحليل اختبار t أظهرت اعلى قوة الالتصاق (SBS). أظهرت MBP,MBEP عدم وجود فرق ذو دلالة إحصائية بينهما. تم استخدام أنوفا في اتجاه واحد واختبار t لتحديد الاختلافات في قوة الالتصاق داخل وبين المجموعات. وكان الفشل متماسك في الأسمنت الراتنج الغالب مع نتائج أعلى في حين لاصقة ومختلطة مع أقل ومساوية.

ألخلاصة المعالجة السطحية مع Monobond etch & prime له تأثير إيجابي على قوة الالتصاق بين العاج الليثيوم ديسيليكات سيراميك المزجج مع الاسمنت الراتج مقارنة مع Monobond Plus.