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Original research

EFFECTS OF DIFFERENT SURFACE TREATMENTS ON THE BOND STRENGTH OF CAD/CAM RESIN NANO CERAMIC OR CEROMER TO RESIN CEMENT

ABSTRACT

Objectives: The purpose of this study was to evaluate the effects of different surface treatments on the micro-tensile bond strength (MTBS) of two different indirect restoration materials (resin nano ceramic CAD/CAM material [Lava Ultimate, 3M ESPE]; ceromer material [Estenia C&B, Kuraray Medical]).

Materials and Methods: Specimens were prepared from each test material in dimensions of 3×10×10 mm. The specimens were divided into five different surface treatment groups: group 1 (control [C]), no treatment; group 2 (acid etching [A]); group 3 (acid etching + universal adhesive [AA]); group 4 (sandblasting [S]); and group 5 (sandblasting + universal adhesive [SA]). The prepared specimens were cemented to composite parts (Filtek Z250 Universal Restorative, 3M ESPE) of the same size using dual-cure adhesive resin cement (Panavia F2.0, Kuraray Medical). A total, 100 bar-shaped specimens (6×1×1 mm) were cut using a low-speed diamond saw (*n*=10 in each group). The MTBS test was performed in all groups (Shimadzu AG-50 kNG, Kyoto, Japan, 1 mm/min). Data were analyzed using a two-way analysis of variance (ANOVA) and Tukey's multiple comparison tests at a significance level of $p<0.05$.

Results: The MTBS values were significantly influenced by the type of restorative material and surface treatment ($p<0.05$). There were statistically significant differences between the materials and surface treatments procedures ($p<0.05$). For Lava Ultimate and Estenia C&B materials, the highest MTBS value was obtained in the SA surface treatment ($p<0.05$) and the lowest MTBS value was obtained in the control groups ($p<0.05$).

Conclusions: The application of silane-containing universal adhesive material after sandblasting was the ideal surface treatment for both materials.

Keywords: Ceromer, composite resins, tensile strength.

Elçin Sağırkaya[1]
*Ayşe Atay[2]

ORCID IDs of the authors:
E.S. 0000-0002-4863-1296
A.A. 0000-0002-5358-0753

1 Department of Prosthodontics, Faculty of Dentistry, Ordu University, Ordu, Turkey
2 Department of Prosthodontics, Faculty of Dentistry, Altinbas University, Istanbul, Turkey

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***Corresponding Author:**
Department of Prosthodontics, Faculty of Dentistry, Altinbas University, İncirli Avenue, No:11/A, 34147, Bakırkoy, Istanbul, Turkey
Phone: +90 212 7094528 **E-mail:** ayseatay82@hotmail.com, ayse.atay@altinbas.edu.tr

INTRODUCTION

New-generation dental indirect restoration materials with improved physical and mechanical properties are widely used in response to aesthetic and biological demands of patients.[1] With the availability of computer-aided design and manufacturing (CAD/CAM), indirect restorations can be fabricated from ceramics and resin composites, in addition to resin-ceramic hybrid materials, which have been recently introduced to the market.[2] The elastic modulus of ceramic materials containing resin matrix is much closer to dentine than conventional ceramics. They are also more easily machined and repaired than glass ceramics (synthetic lithium disilicate) or polycrystalline ceramics.[3] Resin nano ceramic blocks, such as Lava Ultimate (3M/ESPE, St. Paul, MN, USA), are resin matrix ceramics that contain zirconia/silica nano ceramic particles (80% by weight) embedded in a cross-linked resin matrix (20% by weight).[4,5]

The use of new-generation dental composites with improved physical and mechanical properties in indirect restorations has increased with recent advances in resin chemistry.[6] Hybrid resin composites reinforced with nano ceramic fillers known as ceromers (i.e., ceramic-optimized polymers) are used in indirect restorations because of the elastic modulus of these materials is close to dentine tissues, and their wear resistance is similar to a natural tooth.[7] The matrix structure of ceromers is composed of inorganic and organic polymer chains, aliphatic or aromatic dimethacrylate and silicon oxide.[8] The filler component of a ceromer consists of glass and ceramic fillers and a high proportion of silica. The proportion of fillers content in ceromer, which are also referred to as second generation indirect composites, ranges from 70% to 90%.[9]

Effective adhesive bonding minimizes micro-leakage, provides marginal adaptation and increases the fracture strength, thereby enhancing the clinical success of indirect restorations.[10] Mechanical and chemical surface treatments are important to obtain high bond strength between resin ceramic material or ceromer to resin cement.[11] Bonding between the indirect restoration and resin cement is achieved in two ways: by ensuring micro mechanical retention through abrasive surface treatments (e.g. acid etching or sandblasting) of the restorations or by chemical bonding using a silane coupling agent.[12-14] Hydrofluoric acid is the most commonly used chemical agent for modification of the porcelain surface. The acid agent selectively dissolves the glass matrix and the crystalline structure is exposed resulting in the surface of the ceramic becoming rough, which is required for micromechanical retention.[13] Using the sandblasting method, the surface is blasted with aluminum oxide particles to roughen and increase the bonding surface of the restoration material.[14,15] Silane is applied as a bonding agent to improve the bond strength between the indirect restoration and resin cement. The application of silane increased wettability and surface energy by decreasing surface tension.[16] The most commonly used type of silane in dentistry is 3-methacryloyloxy propyl trimethoxysilane.[15,17] ‘Universal’ or ‘multi-mode’ silane-containing adhesive systems that contain a bifunctional monomer have been recently introduced to the market. These silane-containing adhesives enable chemical bonding of ceramic restorations, without the need for a ceramic primer. They can also be used as a bonding agent for dentine and enamel. Therefore, the use of silane-containing adhesive systems decreases the number of operation steps of adhesive cementation.[18]

A number of studies[5,9,11-13,16] have investigated the bond strength of indirect restorative materials to resin cement however there is a lack of literature[16,19,20] comparing the bond strengths of CAD/ CAM resin nano ceramic or ceromer to resin cement. The aim of this study was to evaluate the effects of various surface treatment procedures on the micro-tensile bond strength (MTBS) of two different indirect restoration materials to resin cement. The null hypothesis was that the material types and surface treatment procedures would not affect the bond strength.

MATERIALS AND METHODS

A resin nano ceramic (Lava Ultimate CAD/CAM Restorative, 3M ESPE, St. Paul, MN, USA) and a ceromer (Estenia C&B, Kuraray Medical, Tokyo, Japan) indirect restoration materials were tested in this study and are detailed in Table 1. The specimen preparation design is schematically presented in Figure 1.

Table 1. The brand names, material types, manufacturers and compositions of the materials

Test Materials and Types	Manufacturer	Composition
Lava Ultimate (Resin nano ceramic block)	3M ESPE, St. Paul, MN, USA	80 wt% nanoceramic, 20 wt% resin, silica nanomers (20 nm), zirconia nanomers (4 - 11 nm), nano group particles (0.6 - 10 µm), silane bonding agent
Estenia C&B (Indirect composite resin)	Kuraray Medical Co., Tokyo, Japan	Monomer:Polyurethane methacrylmonomer and mathacrylic acid series monomer) Filler: Surface treated glass powder and surface treated aluminum micro filler) Photocuring catalyst, Colorant and others
Single Bond Universal (Universal Adhesive)	3M ESPE, St. Paul, MN, USA	10- MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane
Panavia F 2.0 (Adhesive resin cement)	Kuraray Medical Co., Tokyo, Japan	Paste A: 10-MDP, silanated silica, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacrylate photoinitiator, and dibenzoyl peroxide Paste B: silanated barium glass, sodium fluoride, sodium aromatic sulfinate, dimethacrylate monomer, and benzoyl peroxide.
Bisco (Porcelain Etchant)	Bisco, Schaumburg, Illinois, USA	9.5% Hydrofluoric Acid Gel
Filtek Z250 (Universal Restorative System)	3M/ESPE, St Paul, Minnesota, ABD	Matrix: BisGMA, BisEMA, UDMA, TEGDMA Filler: Zirconia, silica (82 wt%)

Abbreviations: MDP: 10-methacryloxydecyl dihydrogen phosphate; HEMA: Hydroxyethyl methacrylate; Bis-GMA: bisphenol A-glycidyl methacrylate; Bis-EMA: ethoxylated bisphenol A-glycol dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: Triethyleneglycol Dimethacrylate; BPO, benzoyl peroxide.

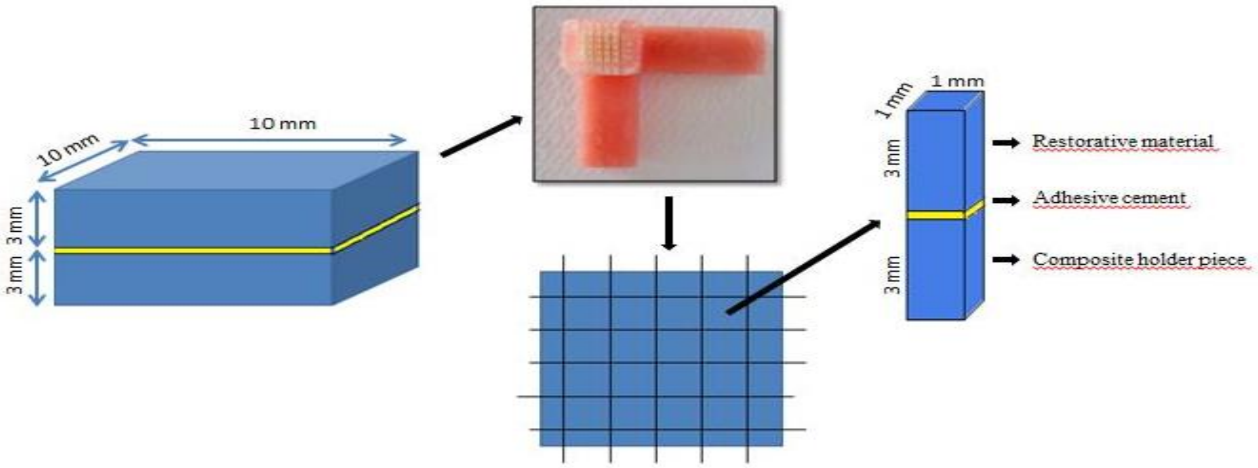

Figure 1. The specimen preparation

Lava Ultimate blocks were cut into 3-mm thick slices using a low-speed diamond saw (Micracut 201, Metkon, Bursa, Turkey). A custom made stainless steel mold was fabricated for the preparation of the specimens from the ceromer material (3×10×10 mm). The ceromer material was applied to the mould in two stages, with each layer allowed to cure for 180 sec in a light and heat curing polymerization unit (CS-110, Kuraray Dental, Osaka, Japan). The specimens were removed from the mold after polymerization and coated with an air-barrier paste (Kuraray Dental) and then cured at 160°C for 15 min. And then they were cleaned in an ultrasonic cleaner (Heatable Ultrasonic Cleaner JP-4820, Skymen, Guangdong, China) at room temperature and then divided into the following five surface treatment groups:

1. Control group (C): No surface treatment was applied.
2. Acid etching group (A): According to the instructions of the manufacturer, 9.5% hydrofluoric acid (Bisco, Schaumburg, IL, USA) was applied for 60 sec to the surface of each sample. After acid etching, the sample was rinsed with pressurized water for 60 sec and dried using an air spray.
3. Acid etching + universal adhesive group (AA): After acid etching, as described above,

Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA) was applied evenly in a thin layer to the cementation surface for 20 sec. According to the manufacturer's instructions, the specimens were air dried for 5 sec and light cured for 10 sec (T Led, Elca Technologies, Imola, Italy).

4. Sandblasting group (S): Sandblast (Renfert GmbH, Hilzingen, Germany) with 50 µm Al_2O_3 at 2.8 bar pressure for 15 seconds at a distance of 10 mm.

5. Sandblasting + universal adhesive group (SA): After sandblasting with 50 µm Al_2O_3 at 2.8 bar pressure for 15 seconds Scotchbond Universal Adhesive was applied evenly to the cementation surface of the specimens for 20 sec, and specimens were air dried for 5 sec and light cured for 10 sec, according to the manufacturer's instructions.

The specimens were bonded to 3×10×10 mm holder pieces prepared from a composite material (Filtek Z250 Universal Restorative, 3M ESPE, St. Paul, MN, USA). The bonding surfaces of the materials were grinded using 600-grit SiC sheet and a dual-cure adhesive resin cement (Panavia F 2.0, Kuraray, Tokyo, Japan) by applying 1 kg of pressure for 5 min in a metal device. After removing the residual cement, the cement application site was covered with an oxygen inhibiting gel (Liquid Strip, Ivoclar Vivadent, Schaan, Liechtenstein) to allow complete polymerization of the resin, and light curing was performed for 40 sec using device with a light output of no less than 550 mW/cm^2 to each surface. The specimens were stored in distilled water at 37°C for 24 h.

An acrylic carrier was prepared as proper for the holder apparatus of the saw to obtain 1 × 1 mm specimens, taken from the 10 ×10 mm specimens (Figure 1). The specimens were bonded to the upper section of the acrylic carrier using cyanoacrylate (Super Bonder Gel, Loctite, Sao Paulo, Brazil). After cutting the specimens, they were detached using a separator at a low speed (3,000 rates/min), first from the acryl carrier and then from each other. The specimens in the outer area were not included in the MTBS test, and the other specimens were checked under a stereomicroscope (M205C, Leica Microsystems, Wetzlar, Germany) at ×10 magnification.

In total, 100 specimens (*n*=10 in each group) were stored in distilled water inside an incubator (Nüve, Istanbul, Turkey) at 37°C for 7 days. The specimens were placed in a metal carrier in the testing device. To avoid any leakage of cyanoacrylate, the bonding area of the material-cement-composite resin was sealed with a thin layer of wax before gluing the specimens to the metal carrier. The MTBS test was performed with a universal testing machine (Shimadzu AG-50 kNG, Kyoto, Japan) at a crosshead speed of 1 mm/min. The results were expressed in megapascal (MPa) values. After MTBS test, the failure modes of specimens were examined under a stereomicroscope (Leica MZ12, Meyer Ins., Bannockburn, IL, USA) at ×20 magnification and recorded as adhesive (cement-resin nano ceramic/indirect composite), cohesive (inside resin cement) or mix (both adhesive and cohesive) failure type.

Statistical analysis

The statistical analyses were performed using SPSS for Windows (12.0, SPSS Inc, Chicago, IL, USA). Two-way ANOVA and Tukey-HSD multiple comparison tests were used for statistical analyses. In all tests, $p<0.05$ was considered as statistically significant.

RESULTS

Two-way ANOVA revealed that the differences among surface treatments and between the materials were statistically significant ($p<0.05$). There were interactions among surface treatments and the materials ($p<0.05$) (Table 2). The mean MTBS values and differences among the groups are presented in Table 3.

Table 2. Results of two-way ANOVA test

Source	Sum of squares	df	Mean square	F	Sig
Material type	159.567	1	159.567	101.550	.000
Surface treatment	1168.721	4	292.180	185.946	.000
Material type * Surface treatment	73.578	4	18.395	11.706	.000

*Significantly different at p<0.05.

Table 3. Mean and SD values for MTBS (MPa) and distribution of failure modes (adhesive/cohesive/mixed)

	Lava Ultimate		**Estenia C&B**	
	MTBS values (Mean ± SD)	**Failure Rates**	**MTBS values (Mean ± SD)**	**Failure Rates**
Control (C)	9.95 ± 1.14^{Aa}	9/0/1	12.54 ± 1.10^{Ba}	7/0/3
Acid Etching (A)	14.88 ± 0.99^{Ab}	6/1/3	15.93 ± 1.44^{Ab}	4/1/5
Acid Etching + Universal Adhesive (AA)	17.10 ± 0.86^{Ac}	2/4/4	17.34 ± 1.43^{Ab}	4/1/5
Sandblasting (S)	16.12 ± 1.07^{Abc}	5/2/3	20.01 ± 1.15^{Bc}	1/6/3
Sandblasting + Universal Adhesive (SA)	19.26 ± 1.26^{Ad}	1/6/3	24.11 ± 1.82^{Bd}	0/8/2

Capital superscripts correspond the same line, lower case superscripts correspond the same column.
*Significantly different at *p<0.05*.

Surface treatments significantly increased the MTBS of the materials compared to the control group (p<0.05). For Lava Ultimate or Estenia C&B materials, the highest MTBS values were obtained in the SA group and the lowest MTBS values were recorded in the C group (p<0.05). The MTBS values of the C, S and SA groups of the Estenia C&B were significantly higher than Lava Ultimate' groups (p<0.05).

For Lava Ultimate, the differences between S group and A, and AA groups were not statistically significant (p>0.05). The differences among the other groups were statistically significant (p<0.05). For Estenia C&B, there was no significant difference in MTBS values between A and AA groups (p>0.05). The differences among the other groups were statistically significant (p<0.05).

Failure pattern distribution of surface treatments and the materials are presented in Table 3. Adhesive fractures were mostly obtained in the C groups, whereas cohesive and mix fractures were mostly observed in all the groups in which the surface treatment procedures were applied. For both materials, cohesive fractures were mostly observed in the SA groups.

DISCUSSION

There are many factors that affect bond strength of indirect restorations to resin cement such as microstructure of restorative materials, type of cement materials, and chemical composition of silane, surface treatment procedures and cementation procedures.[21,22] It has been shown that micro-mechanical locking and chemical adhesion provides a durable bonding of resin-indirect restoration.[23] The present study evaluated the effects of different surface treatments on the bond strength of two types of indirect restorative materials to resin cement. According to the results of the current study, the null hypothesis that the types of materials and surface treatment procedures would not affect the bond strength of the indirect restorative materials to resin cement was rejected.

Surface treatments, including acid etching (9.5% hydrofluoric acid), sandblasting (50 μm Al_2O_3), application of a universal adhesive (silane containing) and their combinations were applied in the current study. These methods are commonly used for intraoral repair or cementation of indirect restorations.[24] Surface treatments are shown to increase the bond strength of resin to indirect aesthetic restoration materials, which was found similar with the results of the current study.[25,26] Some studies reported a linear relationship between bond strength and the elasticity modulus of the material.[22,27] In the current study, it was found that the MTBS values of the Estenia C&B, which has an elasticity modulus close to that of dentine, was higher than Lava Ultimate.

Hydrofluoric acid etching partially dissolved glassy and polymer phase of the ceramic and created micro-porosities by modifying the surface microstructure, thereby increasing mechanical locking between the surface area of the restoration and adhesive cement.[5,28] Previous studies revealed that 10% HF acid gel treatment had no effect on bond strength of resin based CAD/CAM materials to resin cement.[29] The manufacturer of Lava Ultimate does not recommend roughening with acid etching as a surface treatment. Conversely, Loomans *et al*.[19] confirmed that the use of hydrofluoric etching was effective to increase the bond strengths for both Lava Ultimate and Estenia C&B. Similar to this result, in the current study, the use of hydrofluoric etching significantly increased the MTBS values for both Lava Ultimate and Estenia C&B materials compared to the C groups.

Frankenberger *et al.*[30] suggested sandblasting as an alternative to acid etching. For Lava Ultimate, acid etching has not been recommended by the manufacturer, presumably because of the zirconia nanoparticles in the material. Loomans *et al*.[19] stated that sandblasting had a positive effect on bond strength for Lava Ultimate the effect while for Estenia C&B was not statistically significant. In the contrary, in the current study, sandblasting for Lava Ultimate did not show a significant difference as compared to the A and AA groups, whereas the effect on Estenia C&B showed a statistically significant difference as compared to A and AA groups.

One of the surface treatments used in the current study was Single Bond Universal combination with 9.5% HF acid etching and sandblasting usage. Single Bond Universal is a universal adhesive containing silane has been introduced for use in surface treatments before adhesive cementation of indirect composite or ceramic restorations. The application of silane increases wettability and therefore enabled the formation of covalent bonds between the restorative material and resin cement.[22] Queiroz *et al.*[31] investigated the MTBSs of feldspathic ceramic and two different composites treated with acid etching and different ceramic primers. They reported that the use of silane following hydrofluoric acid etching resulted in higher bond strength. Ikemura *et al*.[32] reported that the addition of a silane-monomer mixture to various dental materials, including ceramic, resulted in high bond strength values. Kömürcüoğlu *et al.*[20] investigated the influence of different surface treatments on four point bending strength (FPBS) of novel CAD/CAM restorative materials to resin cement. They reported that application of silane following acid etching increased the FPBS values of Lava Ultimate. Similar to their result, in the current study, for Lava Ultimate, application of silane following acid etching (AA group) significantly increased the MTBS values compared with the A group, whereas for Estenia C&B, there was no significant difference between A and AA groups.

Previous studies[21,33,34] showed that the application of silane had a positive effect on the bond strength to direct composite restorations, whereas some studies could not find a beneficial effect.[35,36] However, the groups that applied Single Bond Universal after sandblasting showed statistically the highest MTBS among the surface treatment groups for both materials. Estenia C&B showed the higher MTBS values than Lava Ultimate. Based on the results of this study, the application of a universal adhesive following sandblasting can be recommended as an ideal surface treatment method for both materials.

Regarding the fracture types, previous research reported that reduced bond strength values were related to adhesive failure rates.[20,37] In the current study, the control groups with the lowest MTBS values regarding the surface treatment was the group with the highest adhesive failure. Toledano *et al.*[37] reported that mixed and cohesive failures were clinically more acceptable than adhesive failures. Cohesive failure of cement points to favourable bonding condition.[38] In the present study, among the surface treatment groups, the SA groups had the highest MTBS values, and these groups also had the highest rates of cohesive failures. For both materials, the use of a universal adhesive following sandblasting significantly increased both bond strength values and cohesive failure rates.

The current study has some limitations that make it difficult to compare the results directly with those of clinical studies. However, the results of the current study can still act as a guide for clinicians. One limitation was the use of only two types of materials and one type of adhesive resin cement. Similar to some studies using the MTBS test method[28,36,38], the lack of aging procedure may be another limitation of the current study. The use of artificial saliva or thermocycling would ensure closer simulation of clinical conditions. To improve the clinical relevance of the findings, future investigations should be performed using different resin cements, different materials and aging procedures.

CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that the surface treatment procedures increased the MTBS bond strength of Lava Ultimate and Estenia C&B materials to resin cement. Estenia C&B had higher MTBS values than Lava Ultimate. The application of a universal adhesive following sandblasting of Lava Ultimate and Estenia C&B materials results in appropriate bond strength values.

DISCLOSURE STATEMENT

No potential conflict of interest was reported by the authors.

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Farklı Yüzey İşlemlerinin CAD/CAM Rezin Nano Seramik ve Ceromerin Rezin Simana Olan Bağlanma Dayanımı Üzerine Etkileri

ÖZ

Amaç: *Bu çalışmanın amacı, farklı yüzey işlemlerinin, iki farklı indirekt restorasyon materyali (CAD/CAM rezin nano seramik [Lava Ultimate, 3M ESPE]; ceromer [Estenia C&B, Kuraray Medical]) ile rezin siman arasındaki mikro çekme bağ dayanımına (MÇBD) etkilerinin değerlendirilmesidir.* ***Gereçler ve Yöntemler:*** *Her test materyalinden 3x10x10 mm boyutlarında örnekler hazırlandı ve beş farklı yüzey işlem grubuna ayrıldı: Grup 1: herhangi bir işlem uygulanmayan kontrol grubu [C]; Grup 2: Asit uygulanan grup [A]; Grup 3: Asit + Silan içeren adeziv uygulanan grup [AA]; Grup 4: Kumlama uygulanan grup [S]); ve Grup 5: Kumlama + Silan içeren adeziv uygulanan grup [SA]. Örnekler, aynı boyutta hazırlanmış olan kompozite (Filtek Z250 Universal Restorative, 3M ESPE) dual-cure adeziv rezin siman (Panavia F2.0, Kuraray Medical) kullanılarak yapıştırılmıştır. Yapıştırılan bu parçalar düşük hızlı bir elmas separe ile kesilerek 100 adet bar şeklindeki (6×1×1 mm) örnekler elde edildi (n= 10). MÇBD testi tüm örneklere yapıldı (Shimadzu AG-50 kNG, Kyoto, Japan, 1 mm/dk). Veriler, iki yönlü varyans analizi (ANOVA) ve Tukey'in çoklu karşılaştırma testleri kullanılarak p<0,05 anlamlılık düzeyinde analiz edildi.* ***Bulgular:*** *MÇBD değerleri restoratif materyallerin tipi ve yüzey işlemlerinden önemli ölçüde etkilendi (p<0,05). Materyaller ve yüzey işlemleri arasinda istatistiksel olarak anlamlı farklılık bulundu (p<0,05). Lava Ultimate ve Estania C&B materyalleri icin, en yüksek MÇBD değeri SA yüzey işlem grubunda ve en düşük MÇBD değeri kontrol grubundan elde edildi (p<0,05).* ***Sonuçlar:*** *Kumlama sonrası silan içerikli universal adezivin uygulanması, her iki materyal için de ideal yüzey işlemidir.*

Anahtar Kelimeler: *Ceromer, bileşik rezinler, gerilme direnci.*

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