RESEARCH

# Effect of thermal and mechanical load cycling on dentin bond strength of a self-etch resin luting cement

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*Selcuk Dent J, 2017; 4: 36-43* (Doi:10.15311/1441.330615)

Başvuru Tarihi: 15 Ocak 2017
Yayına Kabul Tarihi: 02 Mayıs 2017

## ABSTRACT

**Effect of thermal and mechanical load cycling on dentin bond strength of a self -etch resin luting cement**

**Background:** The aim of this study is to evaluate the effects of cyclic thermal and mechanical loads on dentin bond strength of a self-etch resin luting cement under in vitro conditions.

**Methods:** Inlay cavities were prepared on 18 caries-free human mandibular third molars. Restorations were fabricated by using feldspathic porcelain (Vita VM9). Bonding was achieved by using a dual-cure, self-etch resin luting cement (Clearfil Esthetic Cement). Teeth were then randomly divided into 3 groups: Group I: Control group (no thermal or mechanical cycling). Group II: Thermal cycling (thermocycling for 5.000 cycles). Group III: Mechanical load cycling (1.200.000 cycles). Two I-shape sectioned longitudinal cuts were prepared from each tooth (n:12). Total of thirty-six specimens were subjected to tensile forces at a crosshead speed of 1 mm/min, and the maximum load at fracture was recorded. Fracture sites were observed with a stereomicroscope (Olympus, SZ-PT) to identify the failure mode. One tooth from each group was prepared for SEM analysis and interfaces were observed under SEM (435 VP; Leo SEM Products).

**Results:** One-way ANOVA revealed that there were significant differences among the groups ($p<0.05$). Also, Tukey's HSD analysis showed that the mean MTBS of Group III was significantly lower than other groups ($p<0.05$). The difference between Group I and Group II was not significant ($p>0.05$).

**Conclusion:** In this study the principal failure type was found to be interfacial (adhesive) for all groups. Additionally, mechanical loads were observed to be effective on bonding stability while temperature alterations were not significantly influential.

## KEYWORDS

**Mechanical cycling, porcelain, porselen, resin cement, thermal cycling**

## ÖZ

**Termal ve mekanik yükleme siklusunun self–etch rezin yapıştırma simanının dentine bağlanma dayanımı üzerine etkisi**

**Amaç:** Bu çalışmanın amacı, termal ve mekanik yükleme döngülerinin, bir self – etch rezin yapıştırma simanının dentine bağlanma dayanımını in vitro şartlar altında değerlendirmektir.

**Gereç ve Yöntemler:** Çürüksüz, 18 adet mandibular üçüncü molar diş üzerinde inlay kaviteleri hazırlandı. Restorasyonlar, feldspatik porselen kullanılarak hazırlandı (Vita VM9). Dentine bağlantı bir dual- cure, self- etch rezin yapıştırma simanı (Clearfil Esthetic Cement) kullanılarak sağlandı. Sonrasında dişler rastgele üç gruba ayrıldı: Grup I: Kontrol Grubu (termal veya mekanik siklus yok). Grup II: Termal siklus (5.000 termal siklus). Grup III: Mekanik yükleme siklusu (1.200.000 siklus). Herbir dişten (n:12) uzun eksen doğrultusunda kesiler yapılarak ikişer adet I-şeklinde kesitler elde edildi ve toplamda otuz altı adet örneğe, 1 mm/dk hızla çekme kuvvetleri uygulanarak kırılma sırasındaki maksimum kuvvet ölçüldü. Kırık hattı, ayrılma tipini tanımlamak amacı ile stereomikroskop (Olympus, SZ-PT) ile incelendi. Her bir simantasyon grubundan bir diş SEM (435 VP; Leo SEM Products) analizleri yapılmak üzere hazırlanarak arayüzeyler SEM altında incelendi.

**Bulgular:** One-way ANOVA ile gruplar arasında anlamlı farklılıklar olduğu ortaya konuldu ($p<0.05$). Tukey's HSD analizi, Grup III'te görülen ortalama MTBS değerinin Grup I ve Grup II'den anlamlı derecede düşük olduğunu ortaya koydu ($p<0.05$). Grup I ve Grup II arasında istatistiksel olarak anlamlı fark görülmemiştir ($p>0.05$).

**Sonuç:** Bu çalışmanın sonuçlarına bakılarak, tüm gruplar için gözlenen asıl ayrılma tipinin interfasiyal (adeziv) tip olduğu söylenebilir. Ayrıca, sıcaklık değişimleri önemli derece etkili değil iken, mekanik yüklemenin bağlanma stabilitesi üzerine etkili olduğu görülmektedir.

## ANAHTAR KELİMELER

**Mekanik yükleme, porselen, rezin simant, termal yükleme**

The terms "direct restoration" relates mostly to the in situ placement of resin composites while "indirect restoration" refers to the machined or laboratory made ceramics, both alternatives have technical advantages and disadvantages.[1] To overcome the unfavourable properties of direct composite restorations such as poor color stability, low wear resistance and microleakage, ceramic inlays with better esthetic and mechanical characteristics have been developed for the restoration of posterior teeth.[2-7] During the last two decades, several dental ceramic systems were introduced for the fabrication of inlay and onlay restorations. Subsequently, numerous studies about these materials were published.[8-12] Mostly, mechanical properties such as elastic modulus, flexural strength and fracture toughness are assessed under static

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loading conditions for the initial characterization of materials. In this regard, substantial differences are observed between resin composites and dental ceramics.[1] Glass-rich ceramics, such as feldspathic and leucite based, show flexural strengths similar to resin composites[13-14] whereas the values of fracture toughness in resin composites[15], surpass those of glassrich ceramics[16] used for inlays, onlays and veneering of infrastructures.

One of the crucial factors influencing the clinical success of esthetic indirect restorations is the long term bonding stability between adhesive cementing systems and dental tissue.[17-19] Therefore, a durable bond at the tooth-restoration interface is a fundamental for the long-term success of an adhesive restoration.[20,21] In order to prevent the deterioration of sealing between restorative material and tooth structure, the interface must resist dimensional changes.[22] Even after controlling the effects of polymerization shrinkage, deterioration of the restoration may subsequently occur due to chemical degradation or thermal and mechanical stresses.[23,24] As a result of this deterioration process, “fatigue failure” is a form of failure that occurs due to the microscopic cracks within the structure of the materials which were subjected to dynamic and fluctuating stresses. [25]

Immediate bond effectiveness is adequate to evaluate adhesive ability, whereas long-term clinical trials are the ideal method of assessing the durability of adhesive materials.[26,27] However, long-term clinical trials are costly and demands a lot of organizational effort. Meaningful conclusions can only be drawn if an adequate number of crowns or bridges are incorporated and observed for at least 3–4 years. Therefore, it would be beneficial to have validated laboratory tests that allow a prediction of the clinical performance of dental materials.[28]

In this way, the use of mechanical and thermal cycling would allow for in vitro clinical simulation for evaluation of dental materials.[22-24] Fatigue fracture is form of failure that occurs in structures with microscopic cracks subject to dynamic and fluctuating stresses.[28] Mechanical load cycling is designed to apply an occlusal stress on dental restorations to simulate the masticatory process.[29-31] Whereas thermal variations induce fatigue and these cracks propagate and weaken the restoration.[25]

Different commercial brands of self-etch cements have been introduced into the market. Although recent studies have evaluated the performance of self-etch resin cements, little information is available regarding the long-term dentin bond effectiveness. Thus, the aim of this study was to investigate the effects of in vitro long-term degradation strategies (thermal and mechanical load cycling) on the microtensile bond strength (MTBS) of a self-etch resin cement. The following null hypothesis was tested: the aging methods would not affect the microtensile bond strength.

## MATERIALS AND METHODS

Eighteen freshly-extracted, caries-free human mandibular third molars were selected and stored in distilled water. After disinfection (stored in an aqueous solution of 0.5% chloramine T at 4 °C for up to 30 days.) and removal of remnant soft tissues, the occlusal surfaces were flattened perpendicular to the long axis of the teeth with a slow-speed diamond saw sectioning machine (Isomet Buehler Ltd, Lake Bluff, Ill.). Teeth were embedded into autopolymerizing acylic resin (Bayer Ltd, Newbury, United Kingdom) up to 2 mm the cemento- enamel junction.

### Preparation design

Box-shaped inlay cavities were prepared using an inlay preparation set (Acurata GmbH & Co. KG in Thurmansbang, Bavarian Forest, Germany). Each preparation had a length of 6 mm, a width of 3 mm, a depth of 2 mm, and 6 mm convergence of the walls (Figure 1).

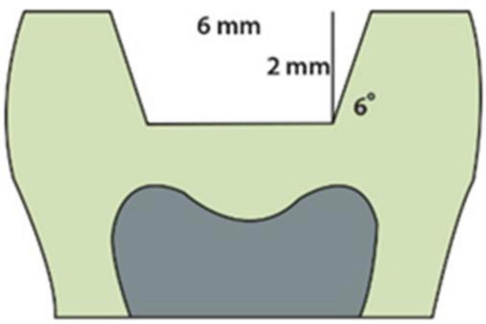


**Figure 1.**

Preparation

### Impression making and fabrication of inlay restorations

Impressions of the 18 prepared teeth were made using vinyl polysiloxane impression material (Virtual, Ivoclar vivadent AG, Schaan, Lichtenstein). To fabricate VİTA VM9 (Vita Zahnfabrik H Rauter GmbH & Co. KG, Bad Sackigen Germany) inlays, refractory dies were duplicated from the polyurethane (Alpha Die MF; Schültz-Dental GmbH, Rosbach, Germany) dies. Ceramic inlays were “built up” on these refractory dies, fired in a porcelain furnace (Vita Vacumat 40 T Vita Zahnfabrik H Rauter GmbH & Co. KG, Bad Sackigen Germany) and glazed, according to the manufacturers’ instructions.

## Cementation procedure

Bonding surfaces of ceramic restorations were etched with 40% phosphoric acid (Ultradent, South Jordan, Utah, USA) for 5 s. The gel was rinsed off with water for 20 s, and then dried with oil-free compressed air. Ceramic primer then applied to bonding surfaces of ceramic and dried sufficiently. ED primer consists of A+B liquids mixed and applied to dentin surfaces and left for 30 seconds and dried.

Dual polymerizing resin cement (Clearfil Esthetic Cement; Kuraray Co Ltd., Osaka, Japan) with an auto-mixing tip was applied to both prepared teeth and adherent surface of the ceramic restorations. Finger pressure was used to stabilize the inlays during bonding to the dentin surface. Excessive cement was removed with an explorer, and the cement was polymerized for 20 s with a high-intensity LED light polymerizing unit (Starlight S Sler, Mectron S.P.A, Genova, Italy) at 1400 mW/cm² (with the light tip to specimen distance of 0 mm). Throughout the experiments, the bonding procedures were carried out in accordance with the manufacturers' instructions. All materials were mixed and applied in a standardized way by the same operator.

## Conditioning the specimens

After cementation, all specimens were stored in distilled water at 37 ℃ for 24 hours. The teeth were then randomly divided into 3 groups:

**Group I:** Control group (no thermal or mechanical cycling).

**Group II:** Thermal cycling (5.000 cycles between 5 and 55 ℃. The dwelling time at each temperature was 30 seconds, and the transfer time from one bath to the other was 2 seconds).

**Group III:** Mechanical load cycling (1.200.000 cycles at an axial force at 1.0 Hz under an 50 N load. 4 mm vertical and 4 mm horizontal movement of the loading tip).

## Microtensile testing

Acrylic resin blocks were mounted in a slow-speed diamond saw sectioning machine (Buehler Isomet 1000 Low Speed Saw, Buehler Ltd, Lake Bluff, IL, USA) with a diamond-rim blade. Each tooth was vertically sectioned both mesial-distally and buccal-lingually along their long axis into 1,2 X 1,2 mm wide sections. Two I-shape–sectioned longitudinal cuts, the top half consisting of ceramic and the bottom half consisting of dentin, were made from each tooth (Figure 2). Therefore there were 12 specimens per group (n:12), and a total of 36 specimens were subjected to tensile forces. These specimens were then attached to the testing apparatus (Harvard Apparatus Co, Inc., Dover, Mass USA) with cyanoacrylate adhesive. The specimens were then subjected to tensile forces at a crosshead speed of 1 mm/ min, and the maximum load at fracture was recorded (in kilograms). Preparation of all specimens and completion of the testing were done by the same operator.

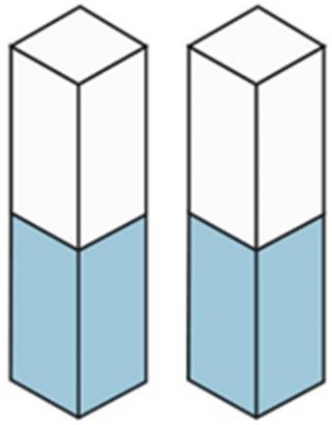

**Figure 2.**

Two I-shape–sectioned longitudinal cuts

## Fracture analysis

After the specimen was tested and removed from the testing apparatus, the fracture sites were observed with a stereomicroscope (Olympus, SZ-PT, Japan) at original magnification X 20 to identify the mode of failure.

The fracture surfaces were classified as follows:

**Mode A:** Adhesive failure between the bonding resin and the ceramic

**Mode B:** Adhesive failure between the bonding resin and dentin

**Mode C:** Cohesive failure

## SEM examination

A tooth from each cementation group was prepared for SEM analysis. After being stored for 24 hours at 37°C, the teeth were sectioned bucco-lingually through the restoration. To observe the interface, the specimens were first polished with 240-, 400-, and 600-grit silicon carbide abrasive paper. The bonding interface was etched with 35% phosphoric acid for 10 seconds and then washed and gently air dried for 3 seconds. Specimens were sputter-coated with gold and interfaces observed under SEM (435 VP; Leo SEM Products, Cambridge, United Kingdom).

### Statistical analysis

Bond strength data were statistically analyzed using one way analysis of variance (ANOVA) and the Tukey's honestly significant differences (HSD) test ($p<.05$). Pretest failures were not included in the statistical analysis. Statistical analyses were performed using a statistical software program (SPSS 20.0 for Windows; SPSS Inc, Chicago, Ill).

## RESULTS

### MTBS Test

Mean MTBS values were recorded for all groups (Table 1). The highest MTBS values were recorded for Group I (control) at 4.35 ± 1.09 N/mm$^2$ and followed by Group II (thermo cyling) at 3.72 ± 0.86 N/mm$^2$ and Group III (mechanical load cycling) at 2.67 ± 0.47 N/mm$^2$. One-way ANOVA has revealed that there were significant differences among the groups ($p<0.05$). Finally Post-Hoc Tukey's HSD analysis showed that; the mean MTBS of Group III was significantly lower than Group I and Group II ($p<0.05$). There were no significant differences between the MTBS of Group I and Group II ($p>0.05$).

### Table 1.

**Mean MTBS values and standard deviations of the groups**

| GROUPS | Mean MTBS (N/mm2)* | ± Std. Deviation (N/mm$^2$) |
|---|---|---|
| Group I | 4.35$^A$ | 1,09 |
| Group II | 3.72$^A$ | 0,86 |
| Group III | 2.67$^B$ | 0,47 |

* Different superscript letters indicate statistically significant differences ($p<0.05$).

### Failure type

The result of this study showed that the principal type of failure was interfacial (adhesive) for all groups, whether between the bonding resin and ceramic (mode A) and adhesive failure between the bonding resin and dentin (mode B).

### SEM Evaluation

SEM images taken from experimental groups revealed that, cracks occurred mostly between resin cement and ceramic (Figure 3–6). The most obvious cracks are presented in fatigue loaded group mostly.

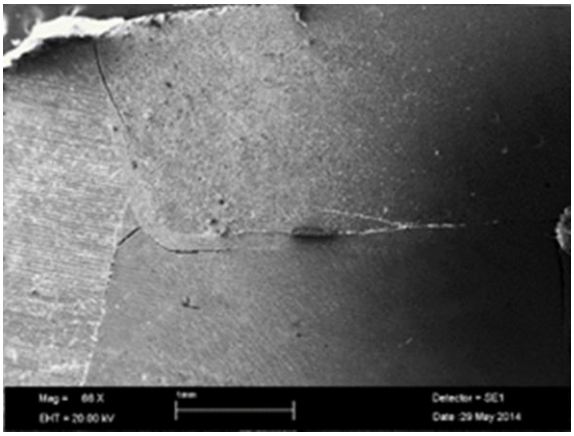

**Figure 3.**

An image from the mechanical loaded group sample (66X)

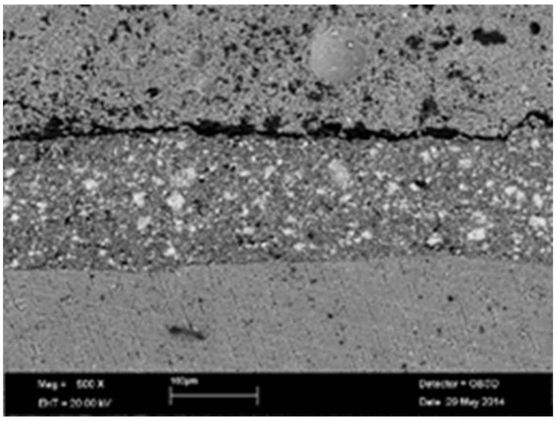

**Figure 4.**

Fatigue loaded group (600X)

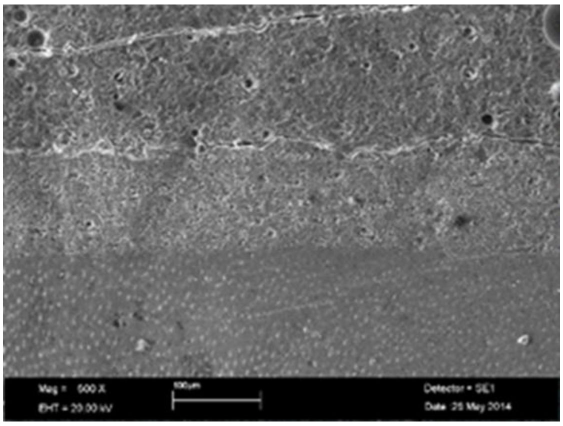

**Figure 5.**

Control group (600X)

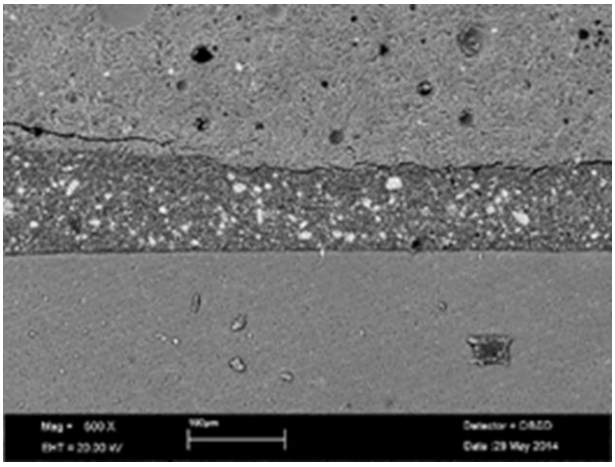


**Figure 6.**

Thermocycled group (600X)

## DISCUSSION

According to the evidence of this study, it can be concluded that the thermal and mechanical cycling loading seem to be both effective on MTBS even the fatigue loading caused significantly higher decrease of MTBS values. Thus, the null hypothesis is rejected.

Dentinal changes can be caused by different water content, pulpal condition before tooth extraction, patient age and composition of dentin. These variations can affect the luting interface between resin cement and dentin.[32] To minimize these effects only human mandibular third molars were used in this study.

Kern et al reported that[33] excessive airborne particle abrasion induced a high loss of ceramic material and is therefore not recommended for cementation of silica-based-all-ceramic restorations. According to these outcomes, sandblasting of the adhesive surfaces was not applied. Also, Shimada *et al*.[34] reported that silane coupling agent application significantly increased the micro-shear bond strengths whether HF or $H_3PO_4$ etching preferred. Moreover, their study showed that the application of 40% phosphoric acid for 5 sec or 60 sec did not show any significant morphological change on the ceramic surface under SEM observation. Depending on these results, Tian *et al*[35] concluded that the acidity is not important but, the role of fluoride on the atomic displacement with silicon dioxide is of greater importance in the etching process.

In the present study, a self- etching resin cement bonding a feldspatic ceramic restoration to dentin was subjected to thermal and mechanical cycling loading separately for evaluating the thermal or mechanical loading effects solely. The resulting MTBS were compared to the control group which was stored in distilled water and was not subjected to long term degradation strategies aiming to separate the effects of wet environment. Still, it must be considered that storage in water may result in hydrolytic degeneration of the interface components, especially of the resin and/or collagen.[32-36]

Also, water can infiltrate and decrease the mechanical properties of the polymer matrix, by swelling and reducing the frictional forces between the polymer chains, a process known as 'plasticization'.[36-38] Furthermore, some interface components, such as uncured monomers and break-down products of previous mechanisms, can elute and so weaken the bond.[39]

Being a widely used aging technique, thermo-cycling was indicated by the ISO TR 11450 standard (1994) as a regimen comprised of 500 cycles in water between 5 and 55°C to be an appropriate artificial aging test. A literature review by Gale *et al*[40] that 10.000 cycles corresponds approximately to 1 year of in vivo functioning, rendering 500 cycles, as proposed by the ISO standard, as being very minimal in mimicking long-term bonding effectiveness.

Also for achieving a statistically relevant number of results, it is recommended that 1.200.000 chewing cycles should be performed to simulate 5 years of clinical service.[41, 42] Proenca *et al*[43] reported that after a 5.000 cycles between 5°C-55°C regimen, specimens treated with HF+silanization showed no tendency but the specimens with no surface treatment showed an increased percentage of debonding. This result can be parallel to our findings till thermal cycling was not found be significantly effective as mechanical cycling did. Still, a decrease of the main MTBS of the thermal cycled group can be defined when compared to control group.

Guarda *et al*[25] demonstrated that there is significant decrease in microtensile bond strength after both thermal (3.000 cycles between 5°C-55°C) and mechanical (100.000 cycles with load of 80 N) cycling loading when lithium disilicate ceramic surfaces treated with 10% HF or $Al_2O_3$ sandblasted. They reported no statistical difference between fatigue and thermocycled groups, which is contrast to our findings. This may be caused by the difference of the loading regimens.

In contrast to the present study, Aguiar *et al*[19], reported that indirect composite resin restorations bonded with Clearfil Esthetic Cement to dentin subjected to fatigue loading showed no difference from their control group. 3 possible explanations can be made for that: First, number of cycles were not enough to make a statement; second, composite resin may have behaved as a shock absorber[44, 19]; third, Clearfil DC Bond application before resin cement may have caused a thick resin layer that may permit direct ion interchange between the cement and dentin interface.

In Nikaido *et al*'s study[44], they compared the effects of C-factor under combined thermal and fatigue cycling loading conditions. On flat dentin surfaces, they reported no significant changes of MTBS after aging regimen; while there were significant decrease in bond strengths with the group of Class I cavity preparations. Also failure types observed to change for the cavity prepared group as adhesive failure while the main failure was cohesive for the flattened dentin surface group. Also Ulker *et al*[45], based on the findings of Nikaido *et al*'s study[44], reported that stresses during cycling loadings can accumulate at the resin-dentin interface, cause the formation of microgaps between adhesive and dentin or plastic deformations in the adhesive itself[46]. These outcomes confirmed our findings.

## CONCLUSION

Within the limitations of this in vitro study, it can be concluded that;

**(1)** Mechanical loading significantly reduces the adhesive performance of self-etched dual cure resin luting cements;

**(2)** Main failure of the fractured sites is mostly adhesive type independent of the aging regimen;

**(3)** Thermal cycling does not effect the durability of the bonding as much as mechanical loading does.

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