



Evaluation of Shear Bond Strength of Different Dental Materials to Bovine Enamel Used as Trauma Splints

Travma Splint Olarak Kullanılan Farklı Dental Materyallerin Makaslama Bağlanma Dayanım Kuvvetinin Değerlendirilmesi

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Abstract

Objective: This research aimed to measure the shear bond strength (SBS) of different dental materials to bovine enamel, while using them as splints for two different time periods.

Materials and Methods: Bovine incisor enamel sections were randomly divided into 10 groups based on test material and surface pretreatment. Each group was further split into 2 subgroups based on the length of the splinting period. Trauma splint materials (Gradia Direct Anterior Composite Resin, Gradia Core, Vertise Flow, Riva Light-Cure) were applied to enamel surfaces using one of two modes of a universal adhesive [etch-and-rinse (E&R) or self-etch (SE)] according to the manufacturers' instructions or with no pre-treatment [self-adhering (SA)]. At the end of splinting period, SBS of samples were evaluated with a universal testing machine, and data was analyzed using one-way ANOVA, post-hoc Tukey's, and paired t-tests.

Results: SBS values for each of the trauma splint materials were significantly higher when used with an E&R protocol as compared to an SE or SA protocol. Moreover, SBS values for Vertise Flow and Riva Light Cure used with an SA protocol were significantly lower than those of all other groups tested ($p < 0.05$).

Conclusion: All the materials tested may be considered appropriate for use as a trauma splint except for Vertise Flow and Riva Light Cure when applied using an SA protocol.

Keywords: Bond strength, dental material, trauma splint

Öz

Amaç: Bu çalışmanın amacı, iki farklı splintleme periyodu sonunda travma splint yapımında kullanılan farklı dental materyallerin sığır minesine makaslama bağlanma dayanım kuvvetini (MBDK) değerlendirmektir.

Gereç ve Yöntemler: Çalışmada taze çekilmiş sığır kesici dişlerinden mine kesitleri elde edildi. Örnekler test materyali, yüzey hazırlık işlemlerine göre rastgele 10 gruba ayrıldı, ardından splintleme süresine göre iki alt gruba ayrıldı. Travma splint materyali olarak dört farklı dental materyal (Gradia Direct Anterior Kompozit Rezın, Gradia Core, Vertise Flow, Riva Light Cure HV) test edildi. Dental materyaller üretici tavsiyeleri dikkate alınarak mine yüzeylerine bir universal adeziv kullanılarak etch&rinse (E&R), self-etch (SE) ve/veya self-adhering (SA) olarak bağlandı. Splintleme sürelerinin sonunda MBDK'ler evrensel test cihazı ile değerlendirildi. Elde edilen verilerin istatistiksel analizde t-testi, tek yönlü ANOVA ve Tukey testleri kullanıldı.

Bulgular: Tüm travma splint materyalleri için E&R modundaki gruplarda diğer uygulama gruplarından daha yüksek MBDK'ler gözlemlendi. En düşük MBDK'ler Vertise Flow ve Riva Light Cure HV'nin SA yani hiç bir yüzey işlemi uygulanmayan gruplarında elde edildi ($p < 0,05$).

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Received/Geliş Tarihi: 02.09.2021

Accepted/Kabul Tarihi: 19.10.2021

*This study was presented as poster presentation with discussion at the 26th International Congress of Turkish Society of Pediatric Dentistry, 10th-13th October 2019.

Sonuç: Vertise Flow ve Riva Light Cure HV'nin hiç bir yüzey işlemi uygulanmayan grupları dışındaki tüm uygulama gruplarının travma splint yapımında kullanılabileceği düşünülmektedir.

Anahtar Kelimeler: Bağlantı kuvveti, dental materyal, travma splinti

Introduction

Traumatic dental injuries are frequently encountered in maxillofacial trauma, and the primary aim of treatment is to preserve the health of the pulp and periodontium (1). Tissue management is crucial to avoid complications (2), and the use of a dental trauma splint (DTS) following tooth repositioning is the standard of care for stabilizing injured teeth and optimizing healing outcomes (3).

From past to present, many different techniques have been used as DTS in dentistry (4,5). Nowadays, The International Association of Dental Traumatology recommends using flexible splints for shorter durations (6), and wire-composite splints are a suitable option for passive mobilization of a traumatized tooth. The splinting materials must be removed from the enamel surface after treatment to prevent bacterial plaque buildup (7,8), without harming the injured tooth or periodontal tissues. Clearly, the high bond strength (BS) to enamel of etch-and-rinse (E&R) adhesives make it hard to remove wire-composite splints without damaging enamel (9). In order to overcome this problem researchers sought alternative splinting systems that had enough BS to endure splinting but were easily removable without damaging the enamel (10,11). Surprisingly, few studies have investigated the effectiveness of dental materials and adhesives in DTS applications, despite the wide range of available products that claim to be versatile and user-friendly. Therefore, this study was conducted to determine the shear BS (SBS) of 4 different dental materials [1 microhybrid composite, 1 dual-cure core, 1 self-adhering (SA) flowable composite (SAFC), 1 resin-modified glass ionomer (RMGIC)] applied to bovine enamel as splints using a universal adhesive (G-Premio Bond) in different modes for splinting periods of 2 different lengths (2 weeks vs 4 months). The tested null hypotheses were as follows: 1) There is no significant difference between the BS of the tested dental materials to bovine enamel; and 2) There is no significant difference between BS of a universal adhesive applied in E&R and self-etch (SE) modes.

Materials and Methods

This study has followed the Checklist for Reporting *In vitro* Studies guidelines for *in vitro* research as discussed in 2014 concept note (12).

Materials

The study was conducted with 4 commercially available dental filling materials: 1 microhybrid composite resin (Gradia Direct Anterior Composite Resin), 1 dual-cure core material (Gradia Core), 1 SAFC (Vertise Flow), 1 RMGIC (Riva

Light Cure HV) and 1 universal adhesive (G-Premio Bond). All materials were applied according to the manufacturers' instructions (Table 1).

Preparation of Enamel Specimens

A total of 34 extracted, non-damaged, permanent bovine incisors were used in this study. Before the experiment, the teeth were polished and were then kept in a 0.1% thymol solution at room temperature. Teeth were examined under a stereomicroscope at $\times 30$ magnification and excluded if they had cracks or stains.

Teeth roots were trimmed and 6 enamel specimens measuring approximately 6x6x3 mm were cut from each tooth, for a total of 200 specimens. These were randomly divided into 10 equal groups (n=20) according to test material and surface pretreatment, and each group was further divided into 2 subgroups (n=10 each) according to length of the immersion period (2 weeks vs 4 months) used to simulate minimum and maximum splinting length (6), as follows:

G/SE: Gradia Direct Anterior Composite Resin and G-Premio Bond in SE mode,

G/E&R: Gradia Direct Anterior Composite Resin and G-Premio Bond in E&R mode,

GC/SE: Gradia Core and G-Premio Bond in SE mode,

GC/E&R: Gradia Core and G-Premio Bond in E&R mode,

VF/SE: Vertise Flow and G-Premio Bond in SE mode,

VF/E&R: Vertise Flow and G-Premio Bond in E&R mode,

VF/SA: Vertise Flow in self adhering (SA) mode (any surface pretreatment)

RLHV/E: Riva Light Cure HV and %37 phosphoric acid

RLHV/PA: Riva Light Cure HV and %20 polyacrylic acid

RLHV/SA: Riva Light Cure HV in SA mode.

G-Power v.3.1.9.2 software (Heinrich Heine, University of Dusseldorf, Dusseldorf, Germany) was used to determine the required minimum sample size according to the data of a previous research (13). Based on the parameters of an alpha-type error of 0.05, a beta power of 0.95, and an effect size of 4.18 the minimal estimated sample size per group was found to be 4.

Enamel surfaces of samples were shear-bond tested by embedding them in acrylic resin cylinders. A cylindrical polyethylene tube was placed on the enamel surface after surface pretreatment, filled with the material to be tested. Specimens were cured with an LED (VALO, Ultradent Products Inc, South Jordan, UT, USA) unit for 20s at

1,000 mV/cm², stored at 37 °C and 100% humidity for 24 hours, and inspected for defects under a stereomicroscope at ×10 magnification. They were then aged in distilled water at 37 °C for 2 weeks or 4 months, with weekly water refreshment.

SBS Testing

SBS was calculated (in MPa) by shearing specimens with a knife-edge blade in a universal testing device (Lloyd LRX, Lloyd Instruments, Fareham, Hants, UK) at a crosshead speed of 1.0 mm/min. Pre-test failures were treated as 0 MPa for group mean calculation. Fractured specimens were examined at ×25 magnification under a microscope (Stemi 2,000-C; Carl Zeiss, Gottingen, Germany) and classified as cohesive (C), adhesive (A), or mixed (M) failures.

Statistical Analysis

One way analysis of variance (ANOVA) was used to detect significant differences ($p < 0.05$) in SBS values among

the groups, and post-hoc comparisons were made using the Tukey HSD test, with the storage period used as the repeated measure. Differences in SBS between subgroups (time periods) of the same group were evaluated using paired t-tests.

Results

Means and standard deviations for the groups are presented in Table 2. The 4-month subgroup of the GC/E&R group had the highest SBS (23.39±7.49), and the 2-week subgroup of the RLHV/SA group had the lowest SBS (0.36±0.15).

In terms of surface pre-treatment, for both splinting periods, the G/E&R and GC/E&R groups had significantly higher ($p < 0.05$) SBS values compared to their counterparts applied in SE mode. SBS values for the VF/E&R group were also significantly higher than those of both the VF/SE and VF/SA groups ($p < 0.05$), whereas SBS values for the RLHV/E and RLHV/P groups were similar ($p > 0.05$) and significantly

Table 1. Composition and application mode of the testing materials

Material	Constituents	Manufacturer	Application
Etch-37 w/BAC	35% phosphoric acid, 1% BAC	Bisco Inc., Schaumburg, USA	1. Gently air dry for 5 s 2. Apply etchant for 15 s 3. Rinse for 10 s and gently air dry for 3 s
G-Premio Bond	10-MDP, phosphoric acid ester monomer, dimethacrylate, 4-MET, MEPS, acetone, silicon dioxide, initiators	GC Corp., Tokyo, Japan	Self - etch mode: 1. Apply the adhesive for 10 s 2. Gently air dry for 5 s 3. Light cure for 10 s Etch - rinse mode: 1. 37% phosphoric acid was applied according to the manufacturer's instructions 2. Apply the adhesive as for the self-etching mode
GC Cavity Conditioner	20% poliacrilic acid, AlCl ₃	GC Corp., Tokyo, Japan	1. Gently air dry for 5 s 2. Apply etchant for 10 s 3. Rinse for 10 s and gently air dry for 3 s
Gradia Direct Anterior Composite	UDMA, dimethacrylate co-monomers, silica (22%), pre-polymerize fillers (size: 0.85 µm, weight: 42%), pigments	GC Corp., Tokyo, Japan	1. Standardized 2-mm-height cylinders were built up on the specimens 2. Light cure for 20 s
Gradia Core	UDMA, dimetacrilat components (1-5%), Fe-III-oxide (Fe ₂ O ₃)	GC Corp., Tokyo, Japan	1. Standardized 2-mm-height cylinders were built up on the specimens 2. Light cure for 20 s
Vertise Flow	GPDM, HEMA, MeHQ, pre-polymerize particles, Ba-glass, SiO ₂ , YbF ₃	Kerr, Orange, CA, USA	1. Standardized 2-mm-height cylinders were built up on the specimens 2. Light cure for 20 s
RIVA Light cure HV	Poliacrilic acid, tartaric acid, HEMA, strontium, fluoroaluminium silicate	SDI, Victoria, Australia	1. Standardized 2-mm-height cylinders were built up on the specimens 2. Light cure for 20 s

BAC: Benzalkonium chloride, MDP: 10-methacryloyloxydecyl-dihydrogen-phosphate, 4-MET: 4-methacryloxyethyltrimellitate anhydride, UDMA: Urethane dimethacrylate, GPDM: Glycerol phosphate dimethacrylate, HEMA: 2-hydroxyethyl methacrylate, MeHQ: Hydrochinon monoethyl ether

higher compared to the RLHV/SA group ($p < 0.05$). No significant SBS decreases were observed over time in any groups. In fact, the SBS values of GC/SE and RLHV/SA groups increased significantly ($p < 0.05$).

Fracture modes for each group are given in Table 2. Most failures were adhesive, followed by mixed and cohesive failures. Adhesive failures were associated with lower BS values.

Discussion

This study found different BS for the different resin materials tested; therefore, the first null hypothesis was rejected. Similarly, the universal adhesive used in different modes (E&R and SE) resulted in different BS; therefore, the second null hypothesis was also rejected.

To avoid the drawbacks of wire-composite splints with E&R adhesive (14), new approaches are required for splinting

traumatized teeth that are easy to implement and have easily removable materials (11). In the present study, 3 different materials were tested as possible alternatives to conventional composite resins.

Composite resin is an advantageous DTS due to its availability (4). Traditional composites bonded with E&R adhesive have high BS (11,15). In fact, there is no consensus regarding the ideal bond-strength for a trauma splint, difficult removal process of composite splints may harm to periodontal healing (14). This study found the conventional microhybrid G/E&R group to have relatively high SBS values (22.85 at 2 weeks and 20.32 at 4 months). Moreover, in line with previous studies (14,16,17), the SBS values of the G/SE group were significantly lower than those of the G/E&R group ($p < 0.05$).

The SBS values of the dual-core GC/E E&R and GC/SE groups were similar to those of the G/E&R and G/SE groups, with the SBS values of the GC/SE group also significantly

Table 2. Mean \pm standard deviations and the fracture modes of groups

Storing criteria					
Groups	2 weeks of water storage at 37 °C		4 months of water storage at 37 °C		p values for paired t-test
	Bond strength (Mean \pm SD)	Mode of failure	Bond strength (Mean \pm SD)	Mode of failure	
G/SE	10.42 \pm 4.27 ^b	A=8 M=2	10.20 \pm 1.37 ^c	A=8 M=2	0.896
G/E&R	22.85 \pm 3.94 ^a	A=3 M=6 C=1	20.32 \pm 4.90 ^{a,b}	A=3 M=6 C=1	0.267
GC/SE	8.11 \pm 1.95 ^{a,b}	A=8 M=2	11.04 \pm 2.39 ^c	A=8 M=2	0.028
GC/E&R	19.17 \pm 4.95 ^a	A=5 M=4 C=1	23.39 \pm 7.49 ^a	A=5 M=4 C=1	0.258
VF/SE	4.22 \pm 2.29 ^{b,c}	A=10	3.21 \pm 1.63 ^d	A=10	0.318
VF/E&R	19.69 \pm 7.75 ^a	A=7 M=3	15.80 \pm 6.16 ^{b,c}	A=7 M=3	0.215
VF/SA	0.57 \pm 0.30 ^c	A=10	0.60 \pm 0.30 ^d	A=10	0.745
RLHV/E	10.70 \pm 3.39 ^b	A=8 M=2	12.88 \pm 4.85 ^c	A=8 M=2	0.340
RLHV/PA	12.82 \pm 4.75 ^b	A=10	14.31 \pm 4.13 ^c	A=10	0.513
RLHV/SA	0.36 \pm 0.15 ^c	A=8 M=2	2.18 \pm 0.94 ^d	A=8 M=2	<0.001
	Time (T)	Group (G)	T*G		
P-values	0.426	<0.001	0.129		

*The letters a, b, c indicate the differences between the application groups within the same measurement time

higher than those of the GC/E&R group ($p>0.05$). As a dual-core material, Gradia Core adds a chemical cure to its light-curing properties to ensure full polymerization, and it can be used in conjunction with a universal adhesive. Giovannetti et al. (18) discovered that disrupted light transmission can reduce the bonding potential of light-cured materials. Dual-cure materials can solve this issue through chemical reaction-based polymerization. The study indicate that dual-cure core materials can using for DTS as an alternative to composite resin, as indicated by the SBS values obtained for the GC groups.

SAFC resin is a chair time reducing alternative to traditional composites, as it doesn't require surface preparation steps (19). However, this study found the BS of VF to have prohibitively lower than, both of the G/E&R and GC/E&R groups, at intact enamel when used in SA mode or with a universal adhesive mode. This is line with Sadek et al. (20), reported that despite the good surface adaptation of low viscosity flowable composites, their BS is lower than hybrid composites and core materials. Clinicians should only use SAFC for DTS construction if combined with an adhesive bonding agent.

The use of an orthodontic luting RMGIC as a DTS was recently described by Kahler et al. (11). In light of studies demonstrating that the use of an adhesive as a surface pretreatment didn't improve the quality of RMGIC restorations (21); this study evaluated RMGIC BS with no pretreatment (RLHV/SA) and with two types of acid pretreatment: polyacrylic acid (RLHV/PA) and phosphoric acid (RLHV/E). The SBSs of both acid-prepared RLHV sub-groups were similar and significantly higher than the self-adhesive sub-group. These results align with previous studies (22).

Splint materials must sustain bond durability for up to 4 months, while dental materials have a limited lifespan and may degrade due to physical, chemical, or biological stimuli (23). Non-etheless, the study found no significant reduction in the BS of any materials tested over time.

The mean SBS values in this study aligned with Table 2's fracture surface analysis. Only the G/E&R and GC/E&R groups showed cohesive fractures, possibly because universal adhesives' higher BS was achieved with extra phosphoric acid etching, resulting in mixed and cohesive fractures. Using the universal adhesive in SE mode resulted in mostly adhesive failures, also SA groups VF and RLHV had similar results too in our study. These observations align with previous *in vitro* studies (16,17,23).

It is important to note that in clinical situations, blood and other oral fluids may limit the BS of dental materials to intact enamel. The ideal minimum BS required for a DTS to intact enamel remains unclear. To determine the effectiveness of new methods/materials for trauma splinting in actual clinical settings, clinical trials are necessary.

Conclusion

Considering the restrictions of this study,

- Resin-based composites and dual-cure core materials, with universal adhesive, showed adequate SBS values for a semi-rigid trauma splint in both E&R and SE modes. However, the higher SBS values in E&R modes may difficult splint removal.

- A SAFC lacked sufficient SBS values for use as a trauma splint without surface pre-treatment. But, when used with a universal adhesive in E&R or SE mode, its SBS values increased.

- RMGIC samples didn't have suitable SBS values for use as a trauma splint without surface pre-treatment. However, its SBS values increased after pretreatment with phosphoric or polyacrylic acid.

Acknowledgement

The authors wish to thank Prof. Soner Çankaya from the Department of Sports Management, Faculty of Yaşar Doğu Sports Science of Ondokuz Mayıs University, Turkey, for his kind help with statistical analysis.

Ethics

Ethics Committee Approval: This article does not contain any studies with human participants or animals performed by any of the authors.

Informed Consent: Since the materials used in this study do not related with any patient, informed patient approval was not required.

Peer-review: Externally peer-reviewed.

Authorship Contributions

Surgical and Medical Practices: Z.Ş., Concept: E.Ş.T., Z.Ş., Design: E.Ş.T., Z.Ş., Data Collection or Processing: Z.Ş., Analysis or Interpretation: E.Ş.T., Z.Ş., Literature Search: E.Ş.T., Z.Ş., Writing: E.Ş.T., Z.Ş.

Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: The authors declared that this study received no financial support.

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