RESEARCH

Comparison of different universal adhesives' bond strength to laser-assisted bleached enamel

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ABSTRACT

Comparison of different universal adhesives' bond strength to laser-assisted bleached enamel

Background: The aim of this *in vitro* study was to evaluate the bond strength of several universal adhesives used in different application modes to laser-assisted bleached enamel.

Materials and Methods: Freshly extracted 72 sound bovine incisors were used for the study. The teeth were bleached with 38% of hydrogen peroxide gel that was activated with a Diode Laser (Epic). The Diode Laser with a 949nm wavelength was used at 7 W in continuous mode for 30 s. After bleaching procedure, the teeth were kept in artificial saliva for 2 weeks. The bleached teeth were randomly assigned into three groups according to different universal adhesive systems (n=24); Single Bond Universal, All-Bond Universal and Adhese Universal. All adhesives were applied in two modes: a-etch&rinse; and b- self etch. A cylinder-shaped resin composite was placed on treated surfaces and cured. After storage of specimen in distilled water for 24 h, they were subjected to shear bond strength test. Data were analyzed using two-way ANOVA followed by Bonferroni test ($\alpha = 0.05$).

Results: The highest mean shear bond strength values were obtained in Single Bond Universal group which was used in self-etch mode ($p<0.05$). No difference was observed between All-Bond Universal and Adhese Universal. In etch&rinse application mode, Adhese Universal resulted in significantly lower bond strength values ($p<0.05$) than Single Bond Universal and All-Bond Universal. For all groups, etch&rinse application mode caused significantly higher bond strength values.

Conclusion: The bonding performance of tested universal adhesives at etch&rinse mode after Diode Laser-assisted bleaching to enamel was superior than self-etch mode application.

KEYWORDS

Bleaching, diode laser, universal adhesives

ÖZ

Farklı üniversal adezivlerin lazerle beyazlatılmış mineye bağlanma dayanıklılıklarının karşılaştırılması

Amaç: Bu *in vitro* çalışmanın amacı farklı uygulama şekillerinde universal adezivlerin lazerle beyazlatma sonrası mine üzerine makaslama bağlanma dayanıklılıklarının değerlendirilmesidir.

Gereç ve Yöntemler: Bu çalışmada yeni çekilmiş 72 kesici sığır dişi kullanılmıştır. Dişler, diyot lazerle (Epic) aktive edilen %38'lik hidrojen peroksit jel ile beyazlatılmıştır. Diyot lazer 949 nm dalga boyu ve 7W'lık devamlı modda 30 saniye kullanılmıştır. Beyazlatma sonrası, dişler yapay tükürükte 2 hafta bekletilmiştir. Beyazlatılmış dişler, farklı universal adeziv sistemlere göre rastgele 3 gruba ayrılmıştır (n=24): Single Bond Universal, All-Bond Universal ve Adhese Universal. Tüm adezivler 2 uygulama şeklinde kullanılmıştır: a- etch&rinse (ER); ve b- self etch (SE). Silindir şekilli kompozit rezin beyazlatma uygulanmış yüzeylere yerleştirilmiş ve ışıkla polimerize edilmiştir. Örnekler 24 saat distile suda bekletildikten sonra makaslama bağlanma dayanıklılığı testine maruz bırakılmıştır. Veriler 2 yönlü ANOVA ve Bonferroni testi kullanılarak analiz edilmiştir($\alpha = 0.05$).

Bulgular: Self-etch modda kullanıldıklarında en yüksek kesme bağlanma dayanıklılığı değerleri Single Bond Universal grubunda görülmüştür ($p<0.05$). All-Bond Universal ve Adhese Universal gruplarında istatistiksel olarak anlamlı bir fark görülmemiştir. Etch&rinse uygulama şeklinde Adhese Universal, anlamlı bir şekilde Single Bond Universal ve All-Bond Universal'den daha düşük makaslama bağlanma dayanıklılık değerleri göstermiştir ($p<0.05$). Tüm gruplarda etch&rinse uygulama modu anlamlı bir şekilde daha yüksek makaslama bağlanma dayanıklılık değerleri göstermiştir.

Sonuç: Test edilen universal adezivlerin diyot lazerle beyazlatılmış mineye olan bağlanma dayanıklılıkları etch&rinse uygulama şeklinde self-etch uygulamaya göre daha yüksek değerler göstermiştir.

ANAHTAR KELİMELER

Beyazlatma, diyot lazer, universal adezivler

Esthetic demands have increased among patients, which leads tooth-bleaching therapies to be common treatments for patients to have beautiful smiles. Bleaching is the most conservative non-invasive option with regard to tissue preservation in discolored teeth. Although home bleaching had been in use for several years with satisfying results, in-office bleaching has become much popular because of shorter time requirement for having whiter teeth.[1] Hydrogen peroxide with high concentrations is generally used for in-office bleaching. Degradation of hydrogen peroxide and release of free radicals can be

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accelerated with the use of light, heat and laser. Among the recommended accelerating agents, laser-assisted in-office bleaching has been pointed out as most promising due to less tooth sensitivity and gingival irritation.[2-4] Various lasers such as Erbium: Yttrium Aluminum Garnet laser, Nd:YAG laser and Diode Laser have been used for laser-assisted in-office bleaching.[4-7]

Bleached teeth might further require restoration. The most important factor for the success of a tooth-colored direct restoration is adequate bonding of the material to tooth structure.[8] Investigations of bleaching effects on enamel bond strength of resin composites reported that bleaching applications can adversely affected their bonding.[9, 10] Therefore a waiting time is crucial between the bleaching treatment and restorative procedures. Time is required for the elimination of residual oxygen from the teeth surface to return the condition that leads to adequate enamel bond strength.[11] At this point, the proper selection of dentin adhesive type might have importance on bonding to bleached enamel.[12-14]

Rapid advancements in adhesive technology during the last years have led dentin adhesives to be applied with fewer steps. In self-etch adhesives, the acid-etching step is no more required. They are capable of modifying and penetrating the smear layer and have the ability to simultaneously etch, prime and bond to the tooth tissues.[15] However there are controversial results about their performance on enamel bonding. Most of the studies reported that applications of etch and rinse adhesives demonstrated higher bond strength values though self-etch adhesives demonstrated lower values.[12, 13]

As a new approach, universal adhesives that can be used in multi modes have been marketed recently. The clinician could use universal adhesive in etch-rinse or self-etch mode according to the case. With a single bottle performing two different strategy of bonding, these adhesives are very cost effective.[16]

Although many in vitro studies had been conducted to assess the bond strength of universal adhesives[17-21], to the extent of authors' knowledge no study has examined the effects of pre-restorative Diode Laser-assisted bleaching on enamel bond strength of universal adhesives. As bleaching has become a routine treatment in clinics, it is important to find out which application mode would enhance the bonding ability of different universal adhesives to laser-bleached enamel. The null hypothesis tested were 1- there would be no difference in bond strength values of any universal adhesives tested; 2- higher bond strength values would be obtained with the use of etch&rinse mode. The aim of this study is to evaluate the enamel bond strength of some recent universal adhesive systems with different application modes after bleaching with Diode Laser.

MATERIALS AND METHODS

Freshly extracted 72 sound bovine incisors were stored in a 0.1% thymol solution for one week at room temperature, followed by storage in distilled water until the time of the experiment. A water-cooled diamond disc (Isomet, Buehler, Lake Bluff, IL, USA) was used to separate crowns from the roots. After examination by means of optical microscope (Leica DM2500, Leica Microsystems, Switzerland) under 10x magnifications for any surface structural damage or defect, all teeth were embedded in a block of acrylic resin with the buccal surface facing up. Enamel surfaces were polished with 200, 400, and 600 grit silicon carbide papers.

A bleaching agent (Laserwhite*20; Biolase, CA, USA) containing 38% of hydrogen peroxide was mixed with a 0.2 ml of violet pigment containing activator gel was used in the study. The bleaching agent was applied approximately in 1mm thickness to cover the buccal surfaces of teeth. The Diode Laser (Epic, Biolase, Irwin, CA, USA) with a 949 nm wavelength was used at 7W in continuous mode for 30 s. The tip of the whitening handpiece of the Diode Laser was positioned perpendicular to buccal surfaces that was about 1 mm away from the gel. The application of laser was repeated for a second time and allowed the whitening gel to remain on the teeth for the minimum of 5 minutes after second laser cycle. Afterwards, the samples were kept in artificial saliva for two weeks. The bleached teeth were randomly distributed into 3 groups (n=24) according to different universal adhesive systems; Single Bond Universal-SU (3M, ESPE, MN, USA), All-Bond Universal-ABU (Bisco Inc., Shaumburg, IL, USA) and Adhese Universal-ADU (Ivoclar, Vivadent, Schaan, Liechtenstein) (Table 1). All adhesives were applied in two modes: a- etch&rinse (ER); and b- self etch (SE). Details of the used adhesives are provided in Table 1. In etch&rinse mode subgroups, enamel surfaces were etched for 30 seconds using 37% phosphoric acid (Total Etch, Ivoclar Vivadent, Liechtenstein). The phosphoric acid was rinsed with compressed water for 10 seconds and then dried with air until the etched enamel surfaces appear as chalky white. All adhesives were used according to manufacturer's instructions. Adhesives were cured using a LED light-curing unit (Radii Plus, SDI, Victoria, Australia) in standard mode (440-480 nm).

Following application of the adhesive systems, resin composite material, IPS Empress Direct (Ivoclar, Vivadent, Schaan, Liechtenstein) was inserted to a Teflon tube (3 mm wide and 2 mm high) that was perpendicularly seated against the prepared enamel surfaces. Resin composite was light-cured for 40 seconds using the same light-curing unit. The output

Table 1.

Materials used in the study

Product name / Batch No	Manufacturer	Composition	Application
Single Bond Universal / #587885	3M ESPE, St Paul, MN, USA	MDP phosphate monomers, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, fillers, ethanol, water, initiators, silane	**Etch&rinse mode** Apply phosphoric acid etching gel (37%) to the enamel, leave it in place for 30 seconds, then rinse and dry. Apply bond to the surface using the applicator brush and rub it for 20 seconds. Dry the entire surface with gentle stream of air over the liquid for about 5 seconds until it no longer moves and the solvent has evaporated completely. Light-cure bond with 1200 mW/cm^2 LED for 10sec. **Self-etch mode** Apply bond to the surface using the applicator brush and rub it for 20 seconds. Dry the entire surface by blowing compressed air until a glossy, immobile film layer results. Light-cure bond with 1200 mW/cm^2 LED for 10sec.
All Bond Universal / #1400006251	Bisco Inc., Shaumburg, IL, USA	MDP, bis-GMA, HEMA, ethanol, water, initiators	**Etch&rinse mode** Apply phosphoric acid etching gel (37%) to the enamel, leave it in place for 30 seconds, then rinse and dry. Apply two seperate coats of bond to the surface using the applicator brush and rub each coat for 15 seconds (no curing between coats). Dry the entire surface by blowing compressed air until bond does not move Light-cure bond with 1200 mW/cm^2 LED for 10sec. **Self-etch mode** Apply two seperate coats of bond to the surface using the applicator brush and rub each coat for 15 seconds (no curing between coats). Dry the entire surface by blowing compressed air until bond does not move Light-cure bond with 1200 mW/cm^2 LED for 10seconds.
Adhese Universal / #V13743	Ivoclar Vivadent, Schaan, Lichtenstein	MDP, Bis-GMA, HEMA, MCAP, decandiol dimethacrylate, dimethacrylate, ethanol, water, initiator, stabilizers, silicon dioxide	**Etch&rinse mode** Apply phosphoric acid etching gel (37%) to the enamel, leave it in place for 30 seconds, then rinse and dry. Apply bond to the surface using the applicator brush and rub it for 20 seconds. Dry the entire surface sufficiently by blowing compressed air until a glossy, immobile film layer results. Light-cure bond with 1200 mW/cm^2 LED for 10seconds. **Self-etch mode** Apply bond to the surface using the applicator brush and rub it for 20 seconds. Dry the entire surface by blowing compressed air until a glossy, immobile film layer results. Light-cure bond with 1200 mW/cm^2 LED for 10seconds.

MDP: 10-Methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-Hydroxyethyl methacrylate, Bis-GMA: Bisphenol A diglycidylmethacrylate.

of the light-curing unit was regularly checked with a radiometer (Hilux Curing Light Meter, Benlioğlu Dental, Ankara, Türkiye) to ensure it was 1400 mW/cm^2. The Teflon tube was carefully removed after light curing and the composite rod was checked for air bubbles. Defected specimens with visible air bubbles or gaps at the interface were discarded.

After storage of the specimens in distilled water at 37°C for 24 hours, specimens were debonded using a universal testing machine (Lloyd, LR50K, Fareham, UK) with a crosshead speed of 1.0 mm/minute. Shear bond strength values were calculated by dividing the highest fracture force (N) by the bonded area and recorded in megapascals (MPa). Debonded specimens were examined under a stereomicroscope (Olympus SZX7, Hamburg, Germany) at 20X magnification, and failure modes were recorded as adhesive (between the enamel and bond or composite and bond), cohesive in composite, cohesive within the enamel, or mixed (a combination of adhesive and cohesive failure).[22] The results were subjected to two-way ANOVA followed by Bonferroni correction test for any significant differences ($\alpha = 0.05$). All statistical analyses were carried out with the SPSS 22.0 version.

RESULTS

The mean shear bond strength values (MPa) and standard deviations of the groups are displayed in Table 2. In self-etch mode, Single Bond Universal showed significantly higher shear bond strength values than All-Bond ($p=0.043$) and Adhese Universal ($p=0.01$). No statistically significant differences were observed between All-Bond and Adhese Universal ($p=0.616$).

Table 2.

Mean values and standard deviations of groups (MPa±SD)

Adhesive Systems	Application Mode	Mean Values (MPa±SD)
Single Bond Universal	Self-etch	58.90±23.60
	Etch&rinse	98.10±37.03
All-Bond Universal	Self-etch	36.26 ±11.21
	Etch&rinse	86.73±22.89
Adhese Universal	Self-etch	24.76±10.26
	Etch&rinse	61.15±15.12

In etch&rinse application mode, Adhese Universal (61.15 MPa) showed significantly lower bond strength values than Single Bond Universal (98.10 MPa) ($p<0.001$) and All-Bond Universal (86.73 MPa) ($p=0.018$) (Table 3).

Table 3.

Statisticaldifference values of different adhesive systems

Mode of Application	Adhesive Systems	p values		
		3M Single Bond Universal	All-Bond Universal	Adhese Universal
Self-etch	Single Bond Universal	-	0.043	0.001
	All-Bond Universal	0.043	-	0.616
	Adhese Universal	0.001	0.616	-
Etch&rinse	Single Bond Universal	-	0.631	0.000
	All-Bond Universal	0.631	-	0.018
	Adhese Universal	0.000	0.018	-

When comparing different application modes (etch&rinse vs self-etch) within each group, statistically significant differences were observed for all groups ($p<0.001$) (Table 4). The distribution of the type of failure modes are presented in Table 5. There were no pretest failures in any of the groups. Failure modes of Single Bond Universal group were mostly mixed at both etch&rinse and self-etch application. On the other hand, Adhese Universal group showed 83.3% and 75% of adhesive failure at etch&rinse and self-etch applications, respectively. Besides, All-Bond Universal failures were mostly mixed in etch&rinse and adhesive at self-etch applications.

Table 4.

Statistical difference values of different application modes

Adhesive Systems	Mode of Application	p values
Single Bond Universal	Self-etch vs Etch&rinse	0.000
All-Bond Universal	Self-etch vs Etch&rinse	0.000
Adhese Universal	Self-etch vs Etch&rinse	0.000

Table 5.

Distribution of failure modes

Groups		Failure Modes		
		Adhesive	Cohesive	Mixed
Single Bond Universal	Etch&rinse	4(%33.3)	-	8(%66)
	Self-etch	4(%33.3)	2(%16.6)	6(%50)
All-Bond Universal	Etch&rinse	5(%41.6)	-	7(%58.3)
	Self-etch	9(%75)	-	3(%25)
Adhese Universal	Etch&rinse	10(%83.3)	1(%8.3)	1(%8,3)
	Self-etch	9(%75)	-	3(%25)

DISCUSSION

The aim of this study was to investigate the bond strength of variable universal adhesives to laser-assisted bleached enamel. Several studies have been conducted to determine the effect of bleaching on bond strength of resin restoratives using different types of adhesive systems.[23,24] Gurgan et al[24] found that etch & rinse adhesive systems demonstrated higher bond strength values than self-etch adhesive systems after bleaching treatments. Similarly, in another study it was shown immediately after bleaching, etch & rinse systems provided higher bond strengths than self-etch adhesives.[23] Anıl et al[25], evaluated the bond strength of an etch & rinse and a self-etch adhesive system to bleached enamel and reported a decrease in bond strength when self-etch adhesive was used. In another study, shear bond strengths of three different self-etch adhesive systems with different pH values were applied to bleached enamel, and it was reported that the bond strength of the mild self-etch system, Clearfil SE Bond with a pH of 1.9 was higher than the systems with moderate and strong pH values.

Unlike most of the studies on this topic, the present study evaluated universal adhesives. Universal adhesives represent the last generation of adhesives, which can be used with different modes[21,27,28]. Various studies have investigated universal adhesives' enamel bond strength[18,20]. To the best of our knowledge, no study had been performed evaluating the bond strength values to laser-assisted bleached enamel using universal adhesive systems. In the present study, the bond strength of Single Bond Universal was found to be statistically higher than the other tested adhesives, All-Bond Universal and Adhese Universal when used in self-etch mode. Therefore, the first null hypothesis, which suggests that there would be no difference in bond strength values of any universal adhesives tested, was rejected. The high bond strength values obtained with Single Bond Universal could be related with its composition. Adhesive systems used in the study have many common features such as the use of 10-methacryloyloxydecyl dihydrogen phosphate. Nevertheless, Single Bond Universal adhesive contains vitrebond copolymer that provides chemical bonding to hydroxyapatite which might have caused higher bond strength results at self-etch mode than other adhesive systems.[29] Suziki et al[30], compared the enamel bond durability of several universal adhesives in different etching modes and similar to our findings, Single Bond Universal had higher bonding values than All-Bond Universal . On contrary, in another study no differences in shear bond strength values were obtained between these two adhesive systems.[31] Imai et al[18] determined the effect of different application and etching methods of different universal adhesives on enamel bond strength. Using the same universal adhesives that had been investigated in the present study, they found no difference between the adhesives' bonding values. Additionally, another study demonstrated similar shear bond strengths for Single Bond Universal and Adhese Universal at etch & rinse and self-etch modes, oppositely to the present studies results.[32] In the literature, SU seems to have different results when compared to an adhesive containing MDP but not polyalkenoic acid copolymer.[33] On the other hand, Diniz et al[22] reported that SU had lower bond strength values than another universal adhesive (Futurabond U) containing MDP (10-Methacryloyloxydecyl dihydrogen phosphate) in etch&rinse mode and similar values with a self-etch system (Clearfil SE) in self-etch mode. The diversity of the results could be related with the bleaching procedure. The studies mentioned above had been conducted on non-bleached enamel samples.

In the present study the used adhesive systems have similar pH values and could be classified as ultramild (pH > 2) adhesives.[34,35] Single Bond Universal has a pH value approximately 2.7, All Bond Universal's pH is 3.2 (2.5-3.5) and Adhese Universal's is between 2.5-3.0. Therefore it might have been expected that their interaction with the enamel in etch & rinse mode could be similar related with their acidic capacity. Nevertheless in the current study, their bond strength results differ from each other in both application modes. In previous studies it was mentioned that enamel[22,30] and dentin[28,36] bonding capacity of universal adhesives varies according to trademark.

An *in vitro* study showed that All Bond Universal and Single Bond Universal demonstrated higher shear bond strength values at dentin than enamel in both self-etch and etch&rinse mode. However, dentin etching for universal adhesives did not influence the bond strength.[31] On the other hand, Moritake et al[37] compared the penetration status of the resin tags for Single Bond Universal for both self-etch and etch&rinse mode at dentin and concluded that the resin tag penetration with self-etch mode was much lower than that with etch&rinse mode.

Universal adhesives are claimed to have same performances when either etch&rinse or self-etch modes are used.[17] However in the present study, etch&rinse mode regardless of the used adhesive systems showed better results than self-etch mode in accordance with previous studies.[18,22,30,38,39] Hence the second hypothesis of the present study is accepted. Prior etching ensures a deeper penetration of the self-etch adhesives into the enamel. However in self-etch application modes of universal adhesives, the increase in the surface of enamel which is dependent on the pH of the adhesive, is lower than that obtained with phosphoric acid.[36,40] A study showed that creating micromechanical retention on the enamel surface through phosphoric acid pre-etching might contribute to better resistance of long-term biomechanical loads when using universal adhesives.[41]

Similarly to our findings, Vermelho et al[31] suggested that etch & rinse mode of Single Bond Universal adhesive resulted in superior shear bond values than self-etch mode on enamel. This statement had been confirmed with previous studies[38,39,42] that reported preliminary etching of enamel significantly increase the bond strength. Additionally, another study compared self-etch and etch & rinse mode of a different universal adhesive (G-Bond Plus) clearly indicated that phosphoric-acid etching significantly increased the bond strength for enamel.[43] The results of the current research showed the superiority of etch & rinse mode compared to self-etch mode for all tested universal adhesive systems.

Hydrogen peroxide is the most commonly used bleaching agent, which releases free oxygen radicals to provoke structural changes and lighten the tooth color. Different types of heat and light activation sources are used for catalyzing hydrogen peroxide decomposition and a faster whitening result.[44] The use of laser energy would be desirable because of minimum exposure to hydrogen peroxide and in the minimum number of treatment sessions.[45] Various lasers such as Nd:YAG, Diode Laser and

Erbium:Yttrium Aluminum Garnet laser have been used to improve the bleaching gel's efficacy.[4,7,46] Photo-activated bleaching methods' popularity led to the investigations of different lasers impact on bond strength, therefore an *in vitro* study compared different types of lasers and it was concluded that different sources had no affect on enamel shear bond strength but duration of light irradiation or different activation methods might change the results, thereby different types of lasers still needs to be investigated.[47] Several studies reported no difference between diode and different laser-assisted bleaching in terms of bond strength of orthodontic brackets.[11,48] As Diode Lasers are most commonly used lasers for bleaching with an advantage of their small sizes, portable features with flexible optic fibers[49], we preferred to use Diode Laser in the present study. However we can not directly extrapolate our results to all laser systems as only one type of laser was investigated.

As presence of oxygen in recently bleached teeth prevents resin polymerization, postponing restoring the bleached teeth for one or two weeks is generally essential.[50] Thus, bond strength evaluations were performed after 15 days in the present study. It is crucial to assess adhesive success from a clinical aspect for reliable results. The present study is an in vitro study, so the results obtained from this study should be confirmed with clinical trials using different types of adhesives.

CONCLUSION

Within the limitations of this in vitro study;

1. Single Bond Universal adhesive showed better results in self-etch mode than other evaluated adhesives. In etch&rinse mode applications, Adhese Universal resulted in lower bonding values than other evaluated adhesives.
2. Etch & rinse application mode of the tested all universal adhesives showed significantly higher bond strength values on bleached enamel.

Clinical relevance

The proper selection of universal adhesive and their application modes might have great importance on adequate bonding to Diode Laser-assisted bleached enamel.

Conflict of Interest

The authors have no conflicts of interest relevant to this article.

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