ORIGINAL ARTICLE

Does a reduction of polymerization time and bonding steps affect the bond strength of brackets?

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Abstract High bond strengths are required in order to avoid bracket failure during treatment while brackets should be removable. In addition, chair time should be kept at a minimum. Therefore, the aim of this study was to investigate any differences in bracket's bond strength to enamel by reducing the polymerization time and the steps of bonding procedure. Five hundred teeth were randomly allocated into 20 groups. The groups were established considering the investigated curing units (quartz-tungstenhalogen (QTH) and light-emitting diode (LED), each with two different polymerization times) and the used bonding agents (Clearfil SE Bond, Transbond Plus, Ideal1, iBond, and Transbond XT Primer following acid etching). The brackets were debonded using a shear-peel load and used to calculate the bond strength. The location of adhesive failure was registered by using the modified adhesive remnant index (ARI). The influence of the parameters curing unit, curing time, and bonding agent as well as their interaction products on bond strength showed that the bonding agent

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80336 Munich, Germany
e-mail: epaschos@med.uni-muenchen.de influenced the bond strength most followed by curing time. The parameter curing unit as well as all the generated interaction products of it showed a lower impact. Regarding the ARI, the bonding agent exhibited also the highest influence. Using a LED resulted in comparable bond strengths as the QTH curing device also at shorter exposure times. Additionally, the two-component self-etching primers showed similar bond strengths compared to the acidetching method. Chair time can be reduced by using twocomponent self-etching primers and LED without decrease of bond strength.

Keywords Orthodontic brackets \cdot Bond strength \cdot LED \cdot QTH \cdot Self-etching primer \cdot Adhesive remnant index (ARI)

Introduction

An important aim during orthodontic treatment is to increase patient comfort and to simplify and decrease chair time. Self-etching primers were developed in order to combine work steps to get a less time-consuming procedure that is easier to handle [1, 2]. At the same time, a rapid development of polymerization devices [3–5] took place.

Light-emitting diode (LED) curing devices are considered as a serious competitor for the gold standard quartz– tungsten–halogen (QTH) lights [6, 7]. They are considered to be more reliable and to provide several other positive features such as a long life time, a minimal heat development, and a low electricity consumption [8, 9]. The new generation of LED curing units with a high light intensity above 1,000 mW/cm² has been found to cure composites in half the time of QTH devices [5, 10].

The level of polymerization correlates with the product of the logarithm of light intensity and curing time [11, 12]. Therefore, increasing light intensity within certain limits should lead to shorter exposure time at the same level of polymerization.

Despite these innovations it has to be proven if this timesaving improvements have an effect on bond strength which is highly important for treatment outcome. High bond strengths are required in order to avoid bracket failure during treatment while on the other hand brackets should be removable without any enamel damage [13].

The aim of this study was to investigate the effect of a high-performance LED curing unit on bond strength of orthodontic brackets in comparison with a conventional QTH curing device using two different polymerization times and different types of bonding agents. The null hypothesis was that there is no difference in bracket's bond strength to enamel by reducing the polymerization time and the application steps for bracket bonding.

Materials and methods

Five hundred extracted human molars of the second dentition without microscopically detected cracked surfaces, restorations, or caries, stored according to international standards (ISO 11405/2003), were randomly allocated into 20 groups (25 in each group). The groups were established considering the two curing units, each with two different polymerization times and the five bonding agents.

Only the buccal surfaces of the molars were used for testing. The roots of the teeth were cut off with a water-cooled low-speed diamond saw. All teeth were cleaned with fluoride- and oil-free pumice for 20 s, rinsed, and dried with oil- and moisture-free compressed air.

Orthodontic stainless steel premolar brackets (Victory Series, 3M Unitek, Monrovia, CA, USA) were used for investigating the shear-peel bond strength (average determined surface area= 9.81 mm^2).

The resin composite Transbond XT (3M Unitek) was used for bracket bonding either after conventional etching or after applying a self-etching primer. Two different twocomponent self-etching primers Clearfil SE Bond (Kuraray Medical Inc., Okayama, Japan) and Transbond Plus (3M Unitek), as well as two one-component self-etching primers Ideal1 (GAC International Inc., Bohemia, NY, USA) and iBond (Heraeus Kulzer, Hanau, Germany) were investigated. The self-etching primer application was done as recommended by the manufacturer.

Conventional etching was performed by using 37% phosphoric acid liquid for 30 s (Etching Liquid, 3M Unitek) followed by the application of Transbond XT Light Cure Adhesive Primer (3M Unitek). For bonding with Clearfil SE Bond and Ideal1 the resin composites recommended by the manufacture, Kurasper F (Kuraray) and Ideal1 (GAC),

respectively, were used. In order to achieve a comparable resin layer thickness, each bracket was bonded using 12 mg of resin with 300 g of force applied with a Correx gauge (Haag-Streit, Bern, Switzerland) for 3 s. Excessive resin was carefully removed.

Light curing followed either for totally 20 or 40 s (10 or 20 s mesially and distally on each bracket) with a conventional QTH curing light (ORTHOLUX XT Visible Curing Light, 3M Unitek) or with a LED curing light unit (Ortholux LED Curing Light, 3M Unitek) for 10 or 20 s (5 or 10 s mesially and distally) at a distance of 3 mm and an angle of 45° to the surface. The above chosen short curing times express the recommendation given by the respective light curing device manufacturer.

The required irradiance of the polymerization device (QTH 400 mW/cm², LED 1,000 mW/cm²) was controlled by measuring with a radiometer (Model 100, Demetron Research Corp., Dansbury, CT, USA) before each curing. After bonding, the teeth were stored for 24 h in deionized water at 37°C (ISO 11405) and thermocycled (Willytec, Dental Research Division, Munich, Germany) at 5°C and 55°C for 1,000 cycles for artificial aging. A complete cycle lasted 65 s (dwell time, 30 s; transfer time, 5 s).

Rectangular stainless steel segment wires $(0.018'' \times 0.022'', 3M$ Unitek) were inserted and ligated to the brackets prior to embedding the crown with the acrylic resin (Technovit 4004, Heraeus Kulzer) in fabricated metal rings. This allowed the same horizontal orientation of the nonembedded buccal surface where the brackets were bonded. The archwire segments acted as a guide for placing the brackets parallel to the shear force direction and were useful to minimize deformation of the brackets during debonding.

The brackets were debonded using a shear-peel load (nonvarying distance of 1.5 mm from the bracket base) on a universal testing machine (MCE 2000ST, quick test, Langenfeld, Germany) at a crosshead speed of 0.5 mm/min (ISO 11405). The load was recorded at bond failure and used to calculate the bond strength (1 MPa=1 N/mm²). The location of adhesive failure was determined under ten times magnification using the modified adhesive remnant index (ARI), which included scores from grade 0 to 3 (0 =no composite resin on tooth, 1 <50% resin on tooth, 2 \geq 50% resin on tooth, 3 =100% resin on tooth). In addition, enamel damages were registered by scoring them with grade 4 [14].

The data were analyzed with the statistical software program SPSS 15.0 (SPSS Inc., Chicago, USA). First, the data were tested for normal distribution (Kolmogorov–Smirnov test) and variance homogeneity (Levene test). A one-way analysis of variance (ANOVA, P>0.05) and the post hoc Tukey's test were conducted. The Kruskal–Wallis test was used to verify these results. The influence of the parameters curing unit, curing time, and bonding agent as

well as their interaction products were analyzed in an ANOVA multivariate test. Significance for all statistical tests was predetermined at P < 0.05. Additionally, a Weibull analysis was performed. Within the Weibull statistics, higher values for characteristic strength [σ_0] and especially for Weibull modulus [*m*], which characterizes the scatter in bond strength, are preferred. The σ_0 corresponds to a probability of failure F=63.2%. The higher the *m* is, the more reliable the tested adhesive system.

Results

The results of the bond strength measurements are shown in Table 1. ANOVA indicated significant differences between the groups (P<0.0001).

The bond strengths achieved with the two different polymerization times (10 and 20 s) by using the LED curing unit were not significantly different (P>0.05) within all investigated bonding agents, although higher values for characteristic strength $[\sigma_0]$ were almost always achieved for a longer polymerization time. For a 40-s exposure time using the QTH curing unit, the brackets bonded by using Transbond Plus and Clearfil SE Bond showed significantly higher bond strengths in comparison to the values measured for 20 s polymerisation time (P < 0.05). No significant difference could be detected for the other bonding agents, although the characteristic strength $[\sigma_0]$ was almost always higher for the longer exposure time with the QTH curing unit. Differences regarding mean bond strength where only achieved when comparing the results after short curing by the QTH unit with that after the longer polymerization modus by the LED. However, this was only the case for the teeth that had been treated by the self-etching primer Transbond Plus or iBond.

Both one-component self-etching primers—Ideal1 and iBond—showed for all investigated conditions, with only one exception, lower characteristic bond strengths $[\sigma_0]$ in comparison to the other bonding agents used in this study.

Regarding the ARI scores significant differences were found between the groups (P < 0.0001) with iBond showing the lowest values. Regarding the used curing unit and the applied curing time no significant difference was found within each bonding agent (P < 0.05). By increasing the polymerization time an increase of enamel damage was obvious for both two-component self-etching primers when using LED for curing after the conventional acid-etching method had been used (Table 2). Enamel damage was evident in 89 of the 500 analyzed teeth (17.8%). The damage extent ranged from small enamel alterations to cracks that were visible to the naked eye. Ideal1 showed no cracks and iBond only five of the 89. The bonding agents

	37% phosp	horic acid]	liquid + Tı	37% phosphoric acid liquid + Transbond XT Transbond Plus	Transboi	ad Plus			Clearfil SE Bond	E Bonc	- <u></u> -		Ideal1			.1	iBond		
Light source	QTH		LED		ЧТУ		LED		QTH		LED		QTH		LED		QTH	LED	0
Time	20	40	10	20	20	40	10	20	20	40	10	20	20	40	10	20 2	20 40	0 10	20
Bond strength (MPa)																			
Mean	13.1^{CDE}	15.9^{EF}	$14.8^{\rm EF}$	14.1^{DEF}		$14.8^{\rm EF}$	13.0^{CDE}	$15.5^{\rm EF}$	$9.6^{\rm ABC} 14.8^{\rm EF} 13.0^{\rm CDE} 15.5^{\rm EF} 13.0^{\rm CDE} 16.9^{\rm F} 14.9^{\rm EF} 15.5^{\rm EF} 7.4^{\rm AB} 6.8^{\rm A} 6.3^{\rm A} 6.9^{\rm A} 7.8^{\rm AB} 8.7^{\rm AB} 10.9^{\rm BCD} 10.0^{\rm BCD} 10^{\rm BCD} 10^{$	16.9^{F}	$14.9^{\rm EF}$	$15.5^{\rm EF}$	7.4^{AB}	6.8^{A}	6.3 ^A (6.5 ^A (.9^A 7.	8 ^{AB} 8.7 ⁻	^{AB} 10.9 ^E
SD	3.6	3.8	4.3	4.5	4.4	4.2	2.7	4.7	3.7	2.9	4.6	3.4	2.3	1.6	1.6 1.0 1.9 2.8	1.9 2	8.	4.2 3.3	3.1
Levene test									0.0001										
ANOVA									0.0001										
Kruskal-Wallis test									0.0001										
Weibull parameters																			
σ_0	14.6	17.3	16.7	18.5	11.0	16.6	14.0	17.1 14.5		18.1	18.1 17.5 16.8		8.1	7.8 6.8	, 6.8	7.9 7.9	.9 8.	8.8 9.7	12.0
m	3.6	5.1	3.3	1.4	2.0	3.5	5.5	4.0	3.6	6.8	2.3	5.4	4.5	3.5 7.4	7.4	2.1 2	2.5 2.1	1 3.2	4.1

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Table 2Distribution ofthe modified AdhesiveRemnant Index		Light source	Time		ARI	score	;		
					0	1	2	3	4
	37% phosphoric acid liquid + Transbond XT	QTH	20 s	N	0	11	9	0	5
				%	0	44	36	0	20
			40 s	N	2	10	8	0	5
				%	8	40	32	0	20
		LED	10 s	N	1	9	7	1	7
				%	4	36	28	4	28
			20 s	N	1	11	4	1	8
				%	4	44	16	4	32
	Transbond Plus	QTH	20 s	N	1	6	12	1	5
				%	4	24	48	4	20
			40 s	N	2	6	9	0	8
				%	8	24	36	0	32
		LED	10 s	N	3	6	8	2	6
				%	12	24	32	8	24
			20 s	N	0	3	7	1	14
				%	0	12	28	4	56
	Clearfil SE Bond	QTH	20 s	N	1	13	7	0	4
				%	4	52	28	0	16
			40 s	N	0	6	10	0	9
				%	0	24	40	0	36
		LED	10 s	N	0	9	11	0	5
				%	0	36	44	0	20
			20 s	Ν	1	10	6	0	8
				%	4	40	24	0	32
	Ideal1	QTH	20 s	N	0	0	25	0	0
				%	0	0	100	0	0
			40 s	N	0	0	20	5	0
			10	%	0	0	80	20	0
		LED	10 s	N	0	3	21	1	0
			20	%	0	12	84	4	0
			20 s	N	2	0	23	0	0
	I	OTU	20 s	% N	8	0	92	0	0
	iBond	QTH	20 s	N 0/	14 56	9	1	0	1
			40 ×	% N	56	36	4	0	4
ARI score key: 0, no adhesive			40 s	N 0/	15	8	1	0	1
on tooth		LED	10 ~	% N	60	32	4	0	4
l < 50% adhesive on tooth,		LED	10 s	N %	9 36	12 48	2 8	1 4	1 4
$2 \ge 50\%$ adhesive on tooth, 3 100% adhesive			20 s	70 N	30 7	40 12	° 3	4	4
on tooth, 4 enamel fracture,			20 8	1V %	28	48	12	4	8
N indicates sample size				70	20	-70	12	т	

with most of the cracks showed also higher characteristic bond strengths $[\sigma 0]$ (Table 1).

Only a few teeth without any adhesive residues (N=59)were found after debracketing (ARI score=0). Just about 12 of them belonged to the groups that were mainly responsible for the above mentioned total amount of enamel damage.

The influence of the parameters curing unit, curing time, and bonding agent as well as their interaction products on bond strength and on the amount of residual adhesive were analyzed and are summarized in Table 3. A significant influence of the above mentioned parameters as well as of their interaction products on bond strength was found. The bonding agent

 Table 3
 Influence of the parameters and their interaction products on bond strength (etha square)

Variables	Bond strength	ARI mod
Bonding agent	0.486	0.207
Time	0.058	0.010
Bonding agent × time	0.038	-
Bonding agent × light source	0.030	-
Light source	0.013	-
Bonding agent \times light source \times time	0.023	-
Light source × time	0.011	-

influenced the bond strength most (higher etha square), followed by curing time. The parameter curing unit as well as all the generated interaction products of it showed a lower impact. Regarding the ARI scores only for the single parameters bonding agent and curing time a significant influence was available. However, as indicated by etha square the bonding agent influenced the ARI score stronger.

Discussion

Studies investigating the physical properties of resin-based composites are of interest for the incremental build-up method that is common in restorative dentistry. They have however limited relevance in orthodontics, as the layer of composite used for bonding brackets is very thin and only for a temporary purpose. Rather the bond strength between bracket and teeth and the amount of remaining resin composite on the teeth after debracketing are relevant orthodontic outcomes. These values allowed to compare the effect of different curing times on the used resin composites as well as the achieved values for different self-etching primers in relation to the conventional etch and prime method. The attempt to decrease operation time is mainly managed by decreasing the steps for etching and priming of the enamel and the curing time. These two factors are the only ones that can result in shorter operation times during bracket bonding.

Therefore, the usage of self-etching primers has increased for bonding of orthodontic brackets. Furthermore, the reduced enamel dissolution and therefore the reduced enamel loss, as a result of the shallower etch pattern [2, 15], indicates an essential benefit of this procedure.

The efficacy of using a self-etching primer on behalf of the bond strengths has been shown in various studies [2, 16–21]. Although some of these investigations were carried out using bovine teeth [2, 19], they can, due to their morphological similarity, be extrapolated to human teeth [22].

Within the present study human teeth were used. In order to standardize our methods factors that may have an influence to the results were considered. The brackets were bonded by one experienced operator, using the same force for an equal duration of time in order to achieve exactly the same adhesive layer thickness as mentioned by many authors [20, 23]. With regard to the different loading rates the international standards (ISO 11405/2003) for testing of the adhesion to tooth structures were used. High-velocity debonding forces that represent the velocity of tooth occlusion during mastication [24] are not in accordance with the complexity of clinical bracket failure, particularly with regard to the undesired contact of brackets during occlusion. However, loading of the bracket, not close to the base, has been claimed to be more representative for in vivo loading and ensures a more consistent application of debonding force [25]. Additionally, we considered the storage time and a supplementary interpolated thermocycling before bond strength testing as they have been suggested as potential critical factors in evaluating the effectiveness of an orthodontic bonding adhesive [26, 27] and are essential in order to achieve clinical relevance of in vitro investigations. Since in the present study many teeth in each group (N=25) we used and as five different types of bonding agents were investigated at once, the impact of different conditions and a minor sample size cannot have affected our results.

Regarding the statistical analysis only a few studies in the literature did a Weibull analysis when investigating orthodontic issues. This important analysis is demanded by various authors [23, 28, 29] for the characterization of bond strength.

The achieved bond strengths of the self-etching primers used in this study were within the range of the values that have been described in the literature [16, 27]. The required clinically acceptable bond strength of 6–8 MPa described in the majority of the available studies [30] were even excelled when looking at the results we got from the conventional etching method and that from the two-component self-etching primers Clearfil SE Bond and Transbond Plus.

In the literature, the mean bond strength was however sometimes significantly less than that of the conventional acid-etching method [2, 16–19, 21, 31]. In this study, Clearfil SE Bond and Transbond Plus achieved similar high bond strength to that of the etch and prime method. These results have also been found in other studies [32, 33].

Both one-component self-etching primers Ideal1 and iBond showed significantly lower bond strength than the two two-component self-etching primers and the conventional method used. This is in agreement with recently published studies were iBond [31, 34] or Ideal1 [20, 32, 35, 36] were investigated.

According to polymerization with a high-performance LED curing device the results of this study are in agreement with most of the studies in the literature were no significant differences were found when comparing the bond strengths with that achieved after curing with QTH [10, 37, 38]. In vivo studies confirmed this [6, 7].

When focusing at the curing time some investigations are reporting a significant difference in bond strength between 10 and 20 s LED curing [9] and for both used devices higher bond strengths with increased curing time [10]. In the present study only for two groups (Transbond Plus and Clearfil SE Bond) the minor exposure time with the QTH curing unit showed significantly lower bond strengths than that achieved by the other three modes of curing. That indicates that reducing the curing time with QTH is not always recommendable. For the other bonding agents no significant difference could be detected, although for these groups the characteristic strength [σ_0] was almost higher but not significant for the longer exposure time.

It has been reported that after using the conventional acid-etching technique more adhesive remained on the enamel surface after debonding than after the use of a selfetching primer [15, 39]. Some new studies however do not agree with this statement [31]. Additionally, it has been stated that phosphoric acid etching produces more enamel fractures than self-etching primer treatment [18]. We found that no benefit exist regarding the residual adhesive on enamel after debonding by using self-etching primers. The complete adhesive remained very seldom on the tooth surface. Furthermore, enamel cracks appeared not more frequent in the conventional etching group. The amount of cracks was associated with the bond strength. The onecomponent self-etching primers that showed the lowest bond strengths showed no (Ideal1) or only a few (iBond) enamel cracks. Both two-component self-etching primers caused a similar fracture mode with that registered for the acid-etching method. The amount of detected cracks is in accordance with that given in the literature [40]. The choice of the curing unit did not influence the ARI scores as described in previous studies [10, 37, 38].

Conclusions

- Clinically acceptable bond strengths of 6 to 8 MPa were found for all self-etching primers used in this study.
- The two-component self-etching primers showed similar bond strengths compared to the conventional acidetching method.
- The one-component self-etching primers showed significantly decreased but clinically acceptable bond strengths in comparison to the two-component selfetching primers.
- The use of the LED curing unit resulted in comparable bond strengths with that achieved by using the QTH curing device also at a shorter exposure time.

Conflict of interest The authors declare that they have no conflict of interest.

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