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Bond strength of orthodontic adhesives to dry and saliva-moistened enamel – a comparative *in vitro* study

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ABSTRACT

Purpose: To compare the bond strengths of six different dental adhesives to dry and saliva-moistened enamel.

Design/methodology/approach: One hundred twenty stainless steel brackets were bonded to human mandibular third molars with six materials: *Enlight LC®* (Ormco Corp), *Grengloo®* (Ormco Corp), *Light Bond®* (Reliance Orthodontic Products), *Charisma®* (Heraeus Kulzer), *SmartBond®* (Gestenco), and *Transbond XT®* with *MIP® primer* (3M Unitek). One half of the specimens in each group (n=10) was bonded to dry enamel, while the other half to saliva-moistened enamel. Bond strength testing was performed with a wire loop loading technique after 30-min incubation in artificial saliva. Failure mode was evaluated using adhesive remaining index (ARI).

Findings: No significant differences in bond strength to dry enamel were noted for all composite materials ($p>0.05$). *SmartBond* exhibited significantly greater bond strength to moistened enamel (7.10 ± 1.47 MPa) and comparable with other composite materials to dry enamel. Composite materials demonstrated significantly reduced bond strength to saliva-moistened enamel ($p<0.001$), except for *Transbond MIP*, whose bond strength was not significantly decreased by saliva contamination ($p=0.089$). There was not statistically significant difference between bond strength of *SmartBond* and *Transbond* to saliva contaminated enamel. A higher incidence of cohesive failures was noted for all materials.

Research limitations/implications: Composite light-cured materials provide adequate bond strength to dry enamel. Cyanoacrylate adhesive provides sufficient bond strength only under moist conditions. *Transbond XT+MIP* has sufficient bond strength to either dry or saliva-moistened enamel.

Practical implications: The study evaluated the bonding strength of orthodontic brackets, which is a critical parameter in orthodontics, especially in situations that do not allow for proper isolation from saliva, such as bonding to impacted teeth following surgical exposure.

Originality/value: Bond strengths evaluated with loop loading technique in different study environments would help the orthodontist to choose the most effective adhesive for the specific clinical situation.

Keywords: Bond strength, Adhesives, Orthodontic attachments

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BIOMEDICAL AND DENTAL MATERIALS AND ENGINEERING

1. Introduction

Dental adhesives for direct bonding of orthodontic brackets to tooth surfaces have been developed since late 1970s. Bonding of an orthodontic attachment to the exposed impacted tooth with adequate isolation can be challenging [1]. Multiple debondings, additional surgical interventions and prolonged treatment time are typical frustrating problems associated with the management of the impacted teeth. Most traditional Bis-GMA adhesives are hydrophobic and require dry etched enamel for mechanical adhesion. In an attempt to reduce bond failure rates under difficult moisture conditions, some manufacturers have introduced hydrophilic adhesives with „moisture insensitive primer", while others have developed „moisture active" adhesives [2].

Moisture insensitive primer (*Transbond MIP*, 3M Unitek) is a hydrophilic and alcohol-based bonding agent containing 2-hydroxyethyl methacrylate and polyalkenoate copolymers with carboxylate groups and ethanol [2,3].

A cyanoacrylate adhesive (*SmartBond*, Gestenco) is a „moisture-active" self-curing bonding system that requires neither a primer nor a light-curing. Reduced number of steps during bonding simplifies the procedure [2,4].

There are numerous *in vitro* studies on the bond strength of orthodontic adhesives. Shear bond strength tests, where the load is applied to the adhesive-enamel interface with the use of knife-edge chisel, are the most commonly used method [5, 6, 7]. However, since brackets bonded on impacted teeth are far from the dental arch, a significant component of tensile stresses exists during orthodontic traction [8, 9]. Therefore, the aim of the present study was to compare the bond strengths of six different adhesives to dry and saliva-moistened enamel using a wire loop loading technique. The null hypothesis tested was that the six materials perform equally well in both conditions.

2. Materials and methods

2.1. Specimen preparation

One hundred twenty extracted human impacted mandibular third molars without any cracks, caries, hypoplastic enamel were used. Teeth were stored in a saturated mineral solution (1.5 mM $CaCl_2$, 0.9 mM KH_2PO_4, 150 mM KCl, 1 mM NaN_3, 20 mM TRIS, pH 7.0) at 4^{o}C until use. Each tooth was mounted vertically in polyvinyl chloride (PVC) tubes using a chemically cured acrylic (*Palavit G*, Heraeus Kulzer, Wehrheim, Germany), leaving only the buccal surface of the crowns exposed. The specimens were stored collectively in the artificial saliva for 24 hours (2.7 g/dm^3 porcine gastric mucin, 0.002 g/dm^3 ascorbic acid, 0.030 g/dm^3 glucose, 0.580 g/dm^3 NaCl, 0.170 g/dm^3, $CaCl_2$ x $2H_2O$, 0.160 g/dm^3 NH_4Cl, 1.270 g/dm^3 KCl, 0.160 NaSCN, 0.330 g/dm^3 KH_2PO_4; 0.200 g/dm^3 urea, 0.340 g/dm^3 Na_2HPO_4, pH adjusted to 6.4 by titrating a phosphate buffer to the solution. Afterwards, the buccal surfaces were cleaned and polished with nonfluoridated pumice and rubber prophylactic cups and thoroughly rinsed.

2.2. Bonding protocol

The teeth were randomly divided into 6 groups, each of 20 specimens, to receive brackets bonded with six different adhesive systems:

I. *Enlight LC*® with *Ortho Solo*® primer (Ormco Corp, Glendora, Calif);
II. *Grengloo*® two-way color change adhesive with *Ortho Solo*® primer (Ormco Corp, Glendora, Calif);
III. *Light Bond*® with Light Bond primer (Reliance Orthodontic Products, Itasca, Ill);
IV. *Charisma*® (shade: A2) with *Gluma Self Etch*® (Heraeus Kulzer, Wehrheim, Germany);
V. *SmartBond*® (Gestenco, Gothenburg, Sweden);
VI. *Transbond XT*® with *Transbond MIP*® primer (3M Unitek, Monrovia, Calif).

One half of the specimens in each group (n=10) was bonded in dry conditions, while the other half in wet conditions. A general bonding protocol for „dry condition" subgroups was as follows:

1) oil-free drying (for 5 s);
2) etching for 30 s (35% phosphoric acid gel, 3M Unitek);
3) rinsing (15 s);
4) oil-free drying (5 s);
5) primer application with a microbrush;

6) gentle blow with an airstream to leave a thin uniform layer of a primer;
7) adhesive application the mesh-base of maxillary premolars brackets with 0.022-in slot (*Victory Series*, 3M Unitek) with a mean bracket area of 9.95 mm^2;
8) bracket placement with a firm pressure against the enamel surface;
9) removal of the excess adhesive with dental probe;
10) light curing: 10 s mesially + 10 s distally with light-emitting diode (LED) curing lamp, >850 mW/cm^2, (*G-Light*®, GC America).

In „wet condition" subgroup, one drop of artificial saliva was placed with the use of Pasteur pipette on the buccal tooth surface prior to primer application, and gently blowed.

In group IV, steps 2-4 were omitted, since *Gluma Self Etch*® is a total etch system. In group V, steps 5, 6, 10 were omitted, since *SmartBond* system does not require the use of a primer and light-curing.

All brackets were bonded by one clinician. Specimens were stored in artificial saliva (37°C) for 30 min prior to debonding.

2.3. Bond strength measurement

Bond strength testing was performed in Vertical Motorized Test Stand (MX2, Imada, Japan) equipped with digital force gauge (ZP, Imada, Japan). Specimens were secured in a jig attached to the base plate of the test stand. A stainless steel wire loop (0.8 mm diameter) was attached to the upper grip of the testing unit and engaged between gingival bracket tie wing and bracket base to produce a pulling force parallel to the bracket base in gingivo-occlusal direction at a crosshead speed of 10 mm/min. The maximum load necessary to debond the bracket was recorded in Newtons. Bond strength was calculated by dividing force values by the bracket base area and expressed in MPa (1 N/mm^2).

2.4. Failure mode evaluation

After debonding, the enamel surface was assessed and scored by one operator according to the modified adhesive remaining index (ARI) with the following scoring criteria:
1) 100% of adhesive on enamel surface with an impression the bracket base;
2) > 90% of adhesive on enamel surface;
3) 10-90% of adhesive on enamel surface;
4) < 10% of adhesive on enamel surface;
5) no adhesive on enamel surface [10].

2.5. Statistical analysis

Means, standard deviations and ranges were calculated for the bond strength values. The data were checked for normality with the Shapiro-Wilk test and for variance homogeneity with Levene test. As the data were normally distributed in all groups, two-way ANOVA with Tukey post hoc tests were used to compare bond strengths of 6 different adhesives to enamel under 2 different conditions. Differences among groups were further investigated using Scheffé multiple range test at 0.05 level of significance. ARI scores were tabulated and analysed with the chi-square test. Statistical calculations were performed using *Statistica 9.0* (Statsoft).

3. Results

Means, standard deviations and ranges of bond strength values in all groups are shown in Table 1. Bond strength of the *SmartBond* to dry enamel surface was significantly lower than those for other groups ($p<0.01$).

Statistical analysis indicated no significant differences in bond strength to dry enamel for all composite materials tested ($p>0.05$).

Table 1.
Mean bond strength values, standard deviations and ranges (MPa) of the 6 adhesive systems applied to dry and saliva-moistened enamel surface

Adhesive	Condition	Mean (MPa) ± SD [range]	Condition	Mean ± SD [range]
Enlight LC	dry	7.05 ± 0.74 [5.90 – 8.40] A	saliva	3.95 ± 1.05 [2.60– 7.11] C
Grengloo	dry	7.41 ± 0.80 [5.87 – 8.79] A	saliva	4.00 ± 1.27 [1.90– 5.29] C
Light Bond	dry	6.75 ± 1.13 [4.01 – 8.11] A	saliva	4.17 ± 1.52 [2.19– 7.23] C
Charisma	dry	8.69 ± 1.51 [4.09 – 9.10] A	saliva	5.06 ± 1.44 [3.10 – 7.34] C
SmartBond	dry	3.91 ± 1.07 [2.90 – 6.30] B	saliva	7.10 ± 1.47 [4.97 – 9.91] D
Transbond XT+MIP	dry	8.89 ± 1.36 [5.97 – 10.54] A	saliva	7.97 ± 1.17 [5.23 – 9.41] D

Table 2.
Frequency distribution of the Adhesive Remnant Index (ARI) assessed after debonding for different adhesives and conditions (dry, wet)

Adhesive	ARI scores									
	1		2		3		4		5	
	dry	wet	dry	wet	dry	wet	dry	wet	dry	wet
Enlight LC	0	0	1	1	7	8	2	1	0	0
Grengloo	0	0	1	1	8	8	1	1	0	0
Light Bond	0	0	0	2	9	7	1	1	0	0
Charisma	1	0	1	3	7	7	2	0	0	0
SmartBond	0	0	1	1	9	7	0	2	0	0
Transbond MIP	1	0	0	2	8	7	1	1	0	0

Under wet conditions, *SmartBond* exhibited significantly greater bond strength (7.10 ± 1.47 MPa) and comparable with those of composite materials to dry enamel.

Composite materials demonstrated significantly reduced bond strength to saliva-moistened enamel ($p<0.001$), except for *Transbond MIP*, whose bond strength was not significantly decreased by saliva contamination ($p=0.089$). There was not statistically significant difference between bond strength of *SmartBond* and *Transbond* to saliva contaminated enamel.

Table 2 shows the failure sites frequency in the tested groups Most of the specimens of the six groups had adhesive failures at the bracket-resin interface. No enamel fractures were found. For each material, chi-squre test indicated no significant difference ($p > 0.05$) in the ARI scores between dry and wet conditions. A higher frequency of ARI scores of 3 in all tested groups indicates a more cohesive failure mode.

4. Discussion

The present study revealed that traditional composite adhesives bond equally sufficient to dry enamel, reaching a minimal sufficient bond strength of 6 to 8 MPa for transferring the loads generated an activated archwire to the tooth, as suggested by Reynolds [11].

Our study has also confirmed reduced bonding strength to the saliva-contaminated enamel of most light-cured composite materials. The exception was *Transbond XT* with moisture insensitive primer (MIP) whose bond strength was not significantly decreased by saliva contamination. This is in agreement with findings of Schaneveldt and Foley [12]. Eliades et al. [2] and Littlewood et al. [13] investigated a combination of *Transbond MIP* with other chemical adhesive (Unite, 3M Unitek) and found no improvement in bond strength under moist conditions. This can be explained by a highly hydrophilic nature of MIP primer and possible incompatibility with hydrophobic resin, which impede the diffusion of the liquid primer into the adhesive paste and eventually lead to inadequate polymerization [2]. In our study, the combination of MIP and light-cured *Transbond XT* provided a bond strength, which was not affected by saliva contamination. This could be a result of additional hydrolytic reaction between carboxylate salt complexes, carboxyl groups of the methacrylate polyalkenoic acid copolymer and residual enamel calcium [2]. Moreover, it has been shown that under wet conditions MIP with light cured composite resins (*Transbond XT*) exhibit better bonding strength than with chemically-cured (*Concise*, 3M Unitek) [2,3].

The bond strength of cyanoacrylate (*SmartBond*) to dry enamel was lower than that of other adhesives since its setting reaction is initiated by the contact of isocyanate groups with water [2]. The bonding strength measured in our study could have been enhanced by subsequent immersion in artificial saliva for 30 min. Earlier studies showed contradictory results of cyanoacrylate bond strength. Whereas in Örtendahl and Örtegren [4] *in vitro* study *SmartBond* achieved greater bond strength than the composite adhesives, other studies drew opposite conclusions. Bishara et al. [14] found not significantly different shear bond strength compared with *Transbond XT* after 30 min. Oztoprak et al. [15] revealed significantly lower bond strength of cyanoacrylate adhesive after 72 hours than that of other composite adhesives: *Transbond XT*, *Transbond Plus* (3M, Unitek) and *Assure* (Reliance, Itasca Ill). Klocke et al. [16] found that after 30 min the bond strength of *SmartBond* was only 33.7%, and 24-hour after bonding it was 46.2% when compared with composite adhesive (*Sondhi Rapid Set*, 3M Unitek). Al-Munajed et al. [17] noted significantly more frequent orthodontic buttons failures for cyanoacrylate after 24 hours and 3 months. Nemeth et al. [18] concluded that *SmartBond* does not provide adquate bond strength under either dry or moist condition after 24 hours and 6 months. Orendain and Espinola obtained opposite results, i.e. *SmartBond* has adequate resistance to debonding under dry conditions and insufficient bond strength to enamel contaminated with artificial saliva [19]. One *in vivo* study by Le et al. showed that *SmartBond* had more than 4 times bond failures (55.6%) compared with composite resin adhesive (11.3%, *Light Bond*, Reliance) and concluded that cyanoacrylate is unsuitable for routine bracket bonding [20]. In the clinical setting, the lower bond strength of the cyanoacrylate can be ascribed to the adhesive deterioration and aging in the oral cavity environment, insufficient curing-time and by gap forming between bracket and the tooth surface [2]. It has been recommended to bond attachments as close as possible to the tooth surface because the *SmartBond* cannot fill gaps due to its low density. This problem can occur during bonding of a bracket or buccal tube to teeth with unusual surface anatomy. It can be speculated that this adhesive would perform better with smaller attachments (buttons, cleats).

In the present study *SmartBond* provided comparable bond strength to saliva moistened enamel to the bond strength of composite materials to dry enamel after 30 min. We have chosen this shorter time period since orthodontic attachments are usually loaded in the same visit as bonding. It could be expected that bond strength after 24 h would be higher since the bond strength of orthodontic adhesives has been shown to increase with time [18,21]. However, some studies reported that bond strengths diminish with water immersion over time [22]. The longevity aspect of bond strength in the oral cavity conditions requires further investigation.

5. Conclusions

1. Composite light-cured materials provide adequate bond strength to dry enamel, with exception to *SmartBond*.
2. Under moist conditions, bond strength of most adhesives is reduced except for *SmartBond* and *Transbond XT+MIP*.
3. *Transbond XT+MIP* has sufficient bond strength to either dry or saliva-moistened enamel.

Additional information

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