Acta Odontologica Scandinavica, 2013; 71: 632–637

informa healthcare

ORIGINAL ARTICLE

Improvement of the orthodontic bracket bond strength with pre-heated composite restoratives

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Abstract

Aim. This study aimed to evaluate the effect of pre-heated composite restoratives on the shear bond strength (SBS) of orthodontic brackets. ***Methods.*** The following materials were tested: a microhybrid composite restorative (Filtek Z250), two nanofilled composite restoratives (Filtek Z350 and NT Premium), a nanohybrid composite restorative (Brilliant) and a conventional orthodontic adhesive (Transbond XT). All materials were stored for 1 h in the incubator either at 25°C (room temperature simulation) or 60°C before bonding 100 orthodontic brackets on bovine lower incisors ($n = 10$). One Coat Bond SL and Transbond XT were used to bond the composite restoratives and the Transbond XT adhesive paste, respectively. After storage in distilled water for 24 h, the brackets were subjected to SBS test at a speed of 0.5 mm/min until bracket debonding. The Adhesive Remnant Index (ARI) was assigned to the fractured specimens. Data were analyzed using the one-way ANOVA and the Tukey post-hoc test ($p < 0.05$). The Kruskal-Wallis test was used to compare ARI scores between the groups ($p < 0.05$). ***Results.*** There was no statistically significant difference between the materials at room temperature. Samples bonded with pre-heated materials showed a statistically higher SBS than those bonded with room temperature materials. Samples bonded with the pre-heated orthodontic adhesive showed the highest SBS among all the pre-heated materials. All preheated composite restoratives produced an SBS mean higher than that of Transbond XT stored at room temperature. ***Conclusion.*** The use of pre-heated composite restoratives and orthodontic adhesives might be an alternative approach to bond orthodontic brackets.

Key Words: *composite resin, shear bond strength, adhesion*

Introduction

Since the introduction of the acid-etch bonding technique, the concept of bonding composite to enamel has been utilized in all fields of dentistry, including when it comes to bonding orthodontic brackets [1]. To enable a controlled three-dimensional orthodontic tooth movement, good bond strength between bracket, adhesive and tooth enamel is required. Bond strength should be strong enough to withstand the oral environment conditions, the forces of mastication and the strains generated by treatment mechanics. However, it should not be excessively strong to the point of making bracket removal difficult or damaging the enamel surface [2].

The improvements in light activation have benefitted composites designed for both restorative dentistry and orthodontic bracket bonding. At present, there are several adhesive systems in the market intended specifically for bonding orthodontic appliances and they are scientifically proven to be effective. However, although orthodontic adhesives have a basic formula similar to that of composites commonly used in clinical restorative procedures, they are often commercially available only from suppliers dealing exclusively with orthodontic materials. For this reason, and possibly also because of the higher consistency of these materials, most orthodontists have elected to work exclusively with such

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(Received 9 March 2012; revised 16 Aprilil 2012; accepted 7 May 2012)

ISSN 0001-6357 print/ISSN 1502-3850 online © 2013 Informa Healthcare
DOI: 10.3109/00016357.2012.700067

systems [3]. To broaden the options for orthodontists, the possibility of using traditional composite restoratives has been raised to bond orthodontic brackets. The shear bond strength (SBS) test has been used to evaluate the suitability of bonding brackets with composite restoratives [1,3,4]. Although similar results between composite restoratives and traditional orthodontic adhesives have been achieved [1,3], other authors have reported lower SBS mean values for composite restoratives [4]. In fact, the higher viscosity observed with composite restoratives tends to compromise their ability to adhere to the bracket base [1]. Thus, if composite restoratives were made more flowable to readily adhere to the bracket base, they could be potentially used to bond orthodontic brackets.

A recent method that has been shown to increase the flowability of composite restoratives consists of pre-heating these materials before photoactivation [5–7]. In addition, the pre-heating process can increase the degree of conversion of composite restoratives [6–8], which may improve the mechanical strength of the polymer network, thereby contributing to a more satisfactory SBS.

A considerable number of microhybrid, nanofilled and nanohybrid composite restoratives have been commercially available and they all differ in terms of physical properties [9]. However, there has been a lack of studies evaluating the influence of pre-heating traditional composite restoratives on the SBS of orthodontic brackets. This study aimed to evaluate the SBS of orthodontic brackets when bonded with either pre-heated or at room temperature composite restoratives in comparison to a conventional orthodontic adhesive. The Adhesive Remnant Index (ARI) was also measured. The following null hypotheses were tested: (1) There is no statistically significant difference in the bond strength; and (2) There is no statistically significant difference in the location of the failure site when using the tested materials to bond orthodontic brackets.

Materials and methods

Materials

The following materials were tested: a microhybrid composite restorative (Filtek Z250, 3M ESPE, St. Paul, MN), two nanofilled composite restoratives (Filtek Z350, 3M ESPE, and NT Premium, Coltène/Whaledent, Altstätten, Switzerland), a nanohybrid composite restorative (Brilliant, Coltène/Whaledent) and a conventional orthodontic fixation system (Transbond XT, 3M Unitek, Monrovia, CA). Table I shows further detail on the adhesive materials used in this study.

Sample preparation

Bovine lower central incisors without major staining and enamel defects were obtained from authorized slaughter houses. One hundred newly-extracted teeth ($n = 10$) [10] were selected, cleaned and stored individually at room temperature in a 0.5% thymol solution. This solution was changed every 7 days.

Polyvinyl chloride (PVC) rings (20×20 mm) were fabricated and filled up with liquid polystyrene resin. The roots were completely embedded into the resin right in the center of each ring, leaving only the crowns exposed. In order to standardize the tooth inclination, a geometry acrylic triangle was used to reassure that the middle third of each tooth buccal surface formed a 90° angle with the flat surface of the cured polystyrene resin base. Following this, the PVC rings were detached from the resin and the specimens stored in distilled water.

Adhesive procedures

One syringe containing the orthodontic adhesive and another four syringes, each containing a different composite restorative material, were stored in the oven (incubator) at 25°C. At the same time, another syringe containing the same orthodontic adhesive and other four syringes, each containing a different composite restorative material, were stored at 60°C. All syringes comprising the same material belonged to the same batch and were kept in the oven (incubator) for 1 h before starting the adhesive procedure [11].

A dental prophylaxis with pumice slurry and rubber cups was carried out on the buccal surface of each tooth for 10 s, after which they were rinsed for 30 s with a water spray and dried with oil-free compressed air for the same amount of time. The rubber cups were replaced with new ones every 10 teeth.

The area receiving the brackets was acid-etched for 15 s with 37% phosphoric acid gel (Scotchbond Etchant, 3M ESPE) applied with a syringe, rinsed for 30 s with water and dried for 20 s until obtaining a chalky appearance. The Transbond XT adhesive paste was used together with its own primer, whilst the 2-step etch-and-rinse adhesive system One Coat Bond SL (Coltène/Whaledent) was used in combination with the composite restorative materials. The primer/adhesive system was spread on the acid-etched enamel surface with a microbrush, air-dried for 10 s (20 cm away) and then photoactivated for 10 s with an LED (Coltolux, Coltène/Whaledent).

The upper central incisor brackets (Dental Morelli, batch #1514350, Sorocaba, Brazil) were held with bracket forceps (Starlet, São Paulo, SP) and positioned in the middle of the buccal surface of the crowns. Using the other end of the forceps, a pressure was exerted for 5 s against each bracket in order to

Table I. Materials used in this study.

Classification	Name (manufacturer)	Composition (% by weight)	Batch
Scotchbond Etchant	Scotchbond Etchant (3M ESPE, St Paul, MN)	Water (50–60), phosphoric acid (30–40), poly(vinyl alcohol) (5–15)	9NL
Conventional orthodontic adhesive	Transbond XT (3M Unitek, Monrovia, CA)	Primer: Bis-GMA (45–55), TEGDMA (45–55), 4-(dimethylamino)-benzeneethanol (<0.5), DL-camphorquinone (<0.3), hydroquinone (<0.03) Adhesive paste: Bis-GMA (10–20), Bis-EMA (5–10), silane-treated quartz (70–80), silane-treated silica (<2), diphenyliodonium hexafluorophosphate (<0.2)	N126445 N120715
2-step etch-and-rinse adhesive system	One Coat Bond SL (Coltène/Whaledent, Altstätten, Switzerland)	Methacrylate*, polyalkenoat methacryliert*, water*	N190766B
Microhybrid composite restorative	Filtek Z250 – A2 shade (3M ESPE, St Paul, MN)	Bis-EMA (1–10), UDMA (1–10), Bis-GMA (1–10), TEGDMA (<5), silane-treated ceramic (75–85)	9N115915BR
Nanofilled composite restorative	Filtek Z350 XT – A2 shade (3M ESPE)	UDMA (1–10), Bis-EMA (1–10), Bis-GMA (1–10), TEGDMA (<5), PEGDMA (<5), 2,6-di-tert-butyl-p-cresol (<0.05), silane-treated ceramic (60–80), silane-treated silica (1–10)	N187943
Nanofilled composite restorative	NT Premium – A2 shade (Coltène/Whaledent)	Bis-GMA (1–25), Bis-EMA (1–25), TEGDMA (1–25), photoinitiator*, filler (75)	1101935
Nanohybrid composite restorative	Brilliant NL – A2 shade (Coltène/Whaledent)	Methacrylates (20), dental glass (1–80), amorphous silica (1–80), photoinitiator*	0174947

Bis-EMA, Bisphenol A Polyethylene Glycol Diether Dimethacrylate; Bis-GMA, Bisphenol A-Glycidyl Methacrylate; TEGDMA, Triethylene Glycol Dimethacrilate; PEGDMA, Polyethylene Glycol Dimethacrylate; UDMA, Urethane Dimethacrylate. * Not provided by the manufacturer.

eliminate the excess of composite from between the bracket mesh and the enamel surface. On each tooth, the composite flash was removed with a regular dental probe (SSWhite, Juiz de Fora, MG, Brazil) for no more than 10 s. After removing the flash around the bracket base, the composites/Transbond XT paste were light-cured for 40 s (20 s on mesial and distal), according to the manufacturer's instructions using an LED device (Coltolux, Coltène/Whaledent). Following bracket bonding, the specimens were stored at room temperature in dark plastic containers filled with distilled water for 24 h before undertaking the shearing bond strength test.

Shear bond strength test

The SBS test was performed at the universal testing machine EZ test (Shimadzu Co., Kyoto, Japan) at a speed of 0.5 mm/min until bracket debonding. The machine was calibrated every five tests. Each sample was firmly gripped by a metal clamp in a position favoring the application of a shearing movement parallel to the buccal surface of the crowns. The apex of a triangle made out of stainless steel rectangular wire (0.019" $\times$ 0.025") was attached to the machine while its base was encased under the lower aspect of the brackets to deliver the shearing stress. Shear strength values were obtained in Newtons and divided by the bracket area (14.12 mm^2) in order to calculate the resistance values in megapascal (MPa).

Adhesive remnant index measurement

Following debonding, the adhesive that remained on the tooth was assessed and scored using the modified Adhesive Remmant Index (ARI) according to a previously described method [12]. All teeth were photographed with the Nikon Digital Camera D60 (Nikon Corp., Tokyo, Japan). This camera was coupled to an F2.8 EX DG Macro Lens of 105 mm (Sigma, Kanagawa, Japan) as well as to an electronic flash macro EM-140 DG NA-iTTL (Sigma, Kanagawa, Japan). The system was set to manual mode, shutter speed of 1/200 s, aperture of f/64 and ISO sensitivity of 200. The zoom lens was adjusted to manual focus, full focus mode, with the diaphragm positioned at #32 and a scale distance of 0.0313 while maintaining the manual mode for flash with an intensity of 1/32. The images of the fractured specimens were processed using the Microsoft Office Picture Manager Software to facilitate the identification of the entire enamel surface containing remnants of composite/adhesive. The brightness was decreased by 10% and the contrast was increased by 100%.

One calibrated examiner (kappa = 0.9) analyzed the images in accordance to the following scoring criteria:

- 1 = The entire composite remained, leaving the impression of the bracket base on the tooth surface;
- 2 = More than 90% of the composite remained on the tooth surface;
- 3 = More than 10% but less than 90% of the composite remained on the tooth surface;
- 4 = Less than 10% of the composite remained on the tooth surface; and
- 5 = No composite remained on the tooth surface.

Statistical analysis

Descriptive statistics data including the mean and standard deviation were calculated for each experimental group. One-way analysis of variance (ANOVA) and Tukey mult-comparison tests were used to compare the SBS between the groups. The Kruskal-Wallis test was used to determine significant differences in the ARI scores. The significance level for all statistical tests was set at 5%. All data were entered and analyzed by SAS software 9.1 for Windows (SAS Institute Inc. Cary, NC).

Results

SBS

All multiple comparisons are shown in Table II. The SBS means produced by the adhesive promoters were statistically similar when these materials were photoactivated after being stored at room temperature (25°C). All pre-heated adhesive promoters presented higher SBS means than those stored at 25°C. The conventional orthodontic adhesive (Transbond XT) showed the highest SBS mean of all pre-heated adhesive promoters evaluated. Conversely, the pre-heated nanohybrid composite restorative showed the lowest SBS mean. All pre-heated composite restoratives

Table II. Shear bond strength means (standard deviations) of the materials at different temperatures.

Materials/temperature	SBS mean (SD)
Filtek Z250 (25°C)	6.8 (0.7) a*
Filtek Z350 XT (25°C)	7.1 (1.0) a
NT Premium (25°C)	6.7 (0.8) a
Brilliant NL (25°C)	6.6 (0.9) a
Transbond XT (25°C)	7.2 (1.3) a
Brilliant NL (60°C)	8.0 (1.0) b
Filtek Z250 (60°C)	8.3 (3.0) bc
Filtek Z350 XT (60°C)	8.8 (0.6) bcd
NT Premium (60°C)	8.4 (1.1) cd
Transbond XT (60°C)	9.6 (0.9) d

*Different lowercase letters in the columns indicate statistically significant differences ($p < 0.05$).

tested in the present study showed a higher SBS mean than Transbond XT stored at room temperature.

ARI

The ARI scores are listed in Table III. The Kruskal-Wallis test detected no difference between the experimental groups despite the high frequency of score 1 (the entire composite remained, leaving the impression of the bracket base on the tooth surface) in most groups. None of the fractured specimens showed cohesive fracture at enamel.

Discussion

The first null hypothesis was rejected because there were statistically significant differences between the tested materials. In fact, all the materials pre-heated at 60°C showed an increase in bracket SBS in comparison with their non-heated homologous stored at 25°C. Bracket bond strength can be influenced by several factors when using a resin-based adhesive. First and foremost, bond strength performance is known to be closely related not only to the composition and characteristics of the adhesive material, but also to the complicated interactions among the various materials at the different interfaces [13]. Also, the degree of conversion of orthodontic adhesives is closely related to bracket SBS means [14].

As a matter of fact, the degree of conversion of composites is known to be incomplete at room temperature [15]. On the other hand, higher temperatures are known to reduce viscosity and enhance radical mobility, resulting in additional polymerization and higher conversion. The collision frequency of unreacted active groups and radicals increases at higher curing temperatures that are below the glass transition temperature. Further, as the composite temperature rises, additional free volume builds up. This, in turn, provides increased mobility to trapped radicals, resulting in further conversion [15]. Therefore, it is likely that all resin-based materials tested in the present study had an increased degree of conversion after being pre-heated at 60°C. Moreover, the lower viscosity of pre-heated materials might have favored a higher flow of the material into the base mesh, creating a stronger interaction between the bracket and the composite materials. The decreased viscosity of pre-heated composite restoratives is likely to render them suitable for bonding orthodontic brackets.

Table III. Frequency distribution of the Adhesive Remnant Index (ARI) scores and inter-group comparison.

		ARI scores					
Material	Temperature	1	2	3	4	5	Test
Filtek Z250	25°C	7	3	0	0	0	ns
	60°C	2	1	3	1	3	
Filtek Z350 XT	25°C	4	2	1	3	0	
	60°C	3	1	1	3	2	
NT Premium	25°C	7	2	1	0	0	
	60°C	2	6	2	0	0	
Brilliant NL	25°C	9	1	0	0	0	
	60°C	3	0	4	1	2	
Transbond XT	25°C	4	2	2	1	1	
	60°C	6	1	1	1	1	

ns, non-significant differences were found between the groups according to the Kruskal-Wallis test ($p > 0.05$).

The conventional orthodontic adhesive tested (Transbond XT) showed the highest SBS mean value of all the materials pre-heated at 60°C. Thus, it can be assumed that Transbond XT might have produced a polymer network with enhanced mechanical strength in comparison to the other materials. However, further tests are necessary to confirm this assumption. Transbond XT contains diphenyliodonium hexafluorophosphate, an onion derivate system that has been shown to improve the polymerization rate of experimental resins, leading to a higher degree of conversion [16]. The increased pre-polymerization temperature (60°C) contributed to improve the polymerization provided by the ternary onion derivate system, which probably fostered a higher mechanical strength network in the pre-heated Transbond XT. In this sense, because our traditional composite restoratives do not contain any onion derivate system, it is likely that their polymerization after the pre-heating process was not additionally improved so well as for Transbond XT, thereby explaining their lower SBS means.

On the other hand, materials that had been stored at 25°C did not produce statistically significant SBS means. Despite remarkable differences between the materials concerning chemical composition and filler characteristics, one can presume that a similar degree of conversion and interactions among interfaces might have occurred when they were utilized at their storage temperature (25°C).

It has classically been suggested that a minimum bond strength of 5.9–7.8 MPa is adequate to withstand the demands of orthodontic treatment [17]. All bond strength mean values of the materials used in this study were higher than this minimum requirement. Despite this, bracket debonding did not lead to cohesive fractures at enamel level, allowing for bracket removal without enamel damage. Also, there was no statistically significant difference between the tested materials concerning ARI scores, thereby validating the second null hypothesis.

It is also worth noting that the SBS mean value of all pre-heated composite restoratives was found to be higher than that of Transbond XT, stored at room temperature (Table II). This finding suggests that any of the composite restoratives tested can be used to bond orthodontic brackets, provided that they are pre-heated before photoactivation. This assumption is certainly of interest to the orthodontic field as it broadens the options available to bond orthodontic brackets for clinical and research purposes.

It is important to bear in mind that clinical conditions may differ significantly from an *in vitro* setting. We need to emphasize here that this was an *in vitro* study and that the sample was not subjected to the rigors of the oral environment. For this reason, we suggest to subject the materials to thermal and mechanical tests for a longer period of time in order to evaluate the SBS improvement obtained from pre-heating them. This will degrade their adhesive bonding and simulate both the conditions of the oral cavity and the impact of orthodontic and mastication forces during treatment.

Acknowledgments

The authors are grateful to Vigodent (Rio de Janeiro, RJ, Brazil) for providing the composite restoratives, the adhesive system and the curing-light unit used in this study.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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