

In-vitro evaluation of an experimental method for bonding of orthodontic brackets with self-adhesive resin cements

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ABSTRACT

Background: Self-adhesive resin cements do not require the surface treatment of teeth and are said to release fluoride, which makes them suitable candidates for bonding of orthodontic brackets. The objectives of this study was to investigate the shear bond strength (SBS) of self-adhesive resin cements on etched on non-etched surfaces *in vitro* and to assess their fluoride release features. **Materials and Methods:** Four fluoride-releasing dual-cure self-adhesive resin cements were investigated. For SBS experiment, 135 freshly extracted human maxillary premolars were used and divided into nine groups of 15 teeth. In the control group, brackets were cemented by Transbond XT (3M Unitek, USA), in four groups self-adhesive resin cements were used without acid-etching and in four groups self-adhesive cements were applied on acid-etched surfaces and the brackets were then deboned in shear with a testing machine. Adhesive remnant index (ARI) scores were also calculated. For fluoride release investigation, 6 discs were prepared for each self-adhesive cement. Transbond XT and Fuji Ortho LC (GC, Japan) served as negative and positive control groups, respectively. The fluoride release of each disc into 5 ml of de-ionized water was measured at days 1, 2, 3, 7, 14, 28, and 56 using a fluoride ion-selective electrode connected to an ion analyzer. To prevent cumulative measurements, the storage solutions were changed daily. **Results:** The SBS of brackets cemented with Transbond XT were significantly higher compared to self-adhesives applied on non-etched surfaces ($P < 0.001$). However, when the self-adhesive resin cements were used with enamel etching, no significant differences was found in the SBS compared to Transbond XT, except for Breeze. The comparisons of the ARI scores indicated that bracket failure modes were significantly different between the etched and non-etched groups. All self-adhesive cements released clinically sufficient amounts of fluoride for an extended period of time. **Conclusion:** For the tested cements, the strongest bonds were obtained by enamel acid-etching prior to bracket bonding. All the self-adhesive resin cements had significant long-term fluoride release and could be recommended as suitable fluoride-releasing orthodontic bonding materials.

Key words

Demineralization, fluoride release, orthodontic brackets, self-adhesive resin cements, shear bond strength

INTRODUCTION

Bonding of orthodontic appliances was first introduced in 1965 and since then has revolutionized the face of orthodontic treatment.^[1] However, traditional systems for bonding orthodontic brackets require multiple steps including etching, rinsing, drying, and application of primer before using adhesive resins. This time-consuming process is a drawback and may have negative effects

on bond strength of brackets especially in posterior areas of mouth where limited vision and accessibility is complicated with the lack of proper blood and saliva control.^[2]

Self-adhesive resin cements which have recently been introduced have lower risk of contamination with oral fluids due to fact that some steps such as etching and priming will be eliminated. These cements are dual cure and in comparison with self-cure and light-cure adhesives, provide the advantages of both setting time control and higher polymerization of adhesive under metallic base of the brackets. In addition, another possible advantage of some of these cements is the release of fluoride as claimed by the manufactures, which may reduce the risk of demineralization around the brackets. Furthermore, several of these cements do not contain the toxic bisphenol A in their compositions.^[3,4]

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Self-adhesive cements are claimed to have several advantages including release of fluoride and improved polymerization and bonding strength.^[5] However, these features need to be investigated *in vitro* and *in vivo* prior to their use as bracket cements. Thus, we aimed to evaluate the bond strength of four marketed self-adhesive cements under orthodontic brackets. In the design of this study, we also included groups to assess the shear bond stress of the cements on etched enamel surfaces as recommended by Bishara *et al.*, De Munck *et al.*, and Lin *et al.*,^[6-8] We also determined the *in-vitro* levels of fluoride release from these orthodontic adhesives. To the best of our knowledge, no studies have evaluated the fluoride release and the bond strength of these self-adhesive cements on etched and non-etched surfaced under orthodontic brackets.

MATERIALS AND METHODS

Materials

Four fluoride-releasing dual-cure self-adhesive resin cements (Breeze [Pentron, USA], SET [SDI, Australia], G-CEM [GC, Japan] and WetBond [PulpDent, USA]) were investigated. For shear bond strength (SBS) experiment, these cements were compared with etch and rinse light-cure resin cement, Transbond XT (3M Unitek, USA) as control group. For fluoride release investigation, those fluoride-releasing dual-cure self-adhesive resin cements were compared to an etch and rinse light-cure resin cement, Transbond XT as a negative control group and a resin modified glass-ionomer Fuji Ortho LC (GC, Japan) as a positive control group.

Assessment of SBS

One hundred and thirty five extracted upper premolars with intact enamel surfaces were used in this laboratory study. All teeth had been extracted 1 month prior to this experiment. They were cleaned and stored in 0.1% thymol solution at a temperature of 37°C. Before starting the experiment, enamel surfaces were cleaned with pumice and rubber prophylactic cups for 10 s and then rinsed for 20 s under tap water and air-dried for 5 s.

One hundred and thirty five standard maxillary premolar metallic brackets with 0.018-inch archwire slots were used (Dentaurum, Germany). The surface areas of the bracket bases were determined with a digital caliper with accuracy of 0.01 mm (10.36 mm²).

Teeth were randomly assigned to 9 groups ($n=15$). The control group consisted of an etch-and-rinse control, Transbond-XT light cure adhesive system. In groups 2-9, the following self-adhesive resin cements were used: SET, Breeze, Embrace WetBond, G-CEM.

In the control group, the teeth were etched with 37% phosphoric acid gel (Ivoclar Vivadent, Liechtenstein)

for 15 s. The etchant was applied at the center of the middle third of the buccal surface. The teeth were then thoroughly rinsed for 20 s with water and air-dried for 5 s. A layer of Transbond XT primer was then applied to the etched surface. Transbond XT paste was then applied to the bracket base and placed on the tooth. The bracket was pressed firmly for 15 s. Excess cement was removed with a scaler. The cement was cured with a halogen light (Coltolux 75, Coltene/Whaledent, Switzerland) at 700 mW/cm² for 20 s (10 s from each proximal side). The output power from light-curing unit was monitored during the preparation process by a light meter (APOZA, Taiwan).

For groups 2-5, the brackets were bonded with SET, Breeze, WetBond and G-CEM cements, respectively. Cements were then applied to the bracket, placed on the tooth, and light cured as described.

In groups 6-9, the teeth were etched with 37% phosphoric acid gel for 15 s prior to the application of the cement. The teeth were then thoroughly rinsed for 20 s with water and air-dried for 5 s. Cements were then applied to the bracket, placed on the tooth, and light cured as described. These groups were labeled as Etch + SET, Etch + Breeze, Etch + WetBond and Etch + G-CEM respectively.

All specimens were stored in distilled water at 37°C for 7 days. They were then subjected to 3,000 thermocycles between 5°C and 55°C.

The thermocycled brackets were debonded with a shear load applied in a universal testing machine (Zwick, Germany) with a metal chisel at a crosshead speed of 1 mm/min. The specimens were mounted on the machine and hence that the end of the chisel applied a compressive load directly to the occlusal aspect of bracket-tooth interface parallel to the long axis of the bond interface [Figure 1]. The maximum loads to debond the brackets were recorded in megapascals (MPa).

Adhesive remnant index

Each tooth and its corresponding bracket were viewed with a stereomicroscope (Blue Light Industry USA, CA) and scored according to the ARI. The values for the ARI are as follows: 0: No adhesive left on the tooth; 1: Less than half of the adhesive left on the tooth; 2: More than half of the adhesive left on the tooth; and 3: All adhesive left on the tooth with an impression of the bracket mesh. All tests were performed by the principal author.

Assessment of fluoride release

The method proposed by Cacciafesta *et al.*, was followed to investigate the fluoride resale of our samples.^[9] In summary, samples were created by using silicon molds measuring 8 mm diameter and 2 mm height. After injection of each

adhesive with a syringe, the surface of the samples was covered with a thin plastic layer in order to avoid contact of oxygen with an adhesive layer. The adhesives were cured with a halogen light-curing unit through the glass plates for 40 s (20 s from each side). A total of 36 samples were prepared and six for each adhesive cement. Transbond XT was considered as our negative control group and Fuji Ortho was considered as our positive control group.

Each test specimen was immersed in 5.0 mL of deionized distilled water in a sealed container and stored in an incubator at 37°C. The containers were randomly numbered to enable the fluoride testing to be carried out blindly. The fluoride levels of the solution in which the specimens were immersed were measured by means of a fluoride electrode. As the fluoride electrode is sensitive to changes in pH, total ionic strength adjustment buffer was added to a water specimen before testing to hold its pH between 5.0 and 5.5. As suggested by Cacciafesta *et al.*, the fluoride electrode was calibrated before use. This was carried out by using a series of standard solutions of sodium fluoride of the following PPM concentrations: 1000, 100, 10, 1, and 0.1.^[9]

At days 1, 2, 3, 7, 14, 28, and 56 of the experiment, the specimens were removed from the incubator and the fluoride was measured. The concentrations of fluoride released from each material were recorded and then converted into $\mu\text{g}/\text{cm}^2$ in order to demonstrate the amount of fluoride released per sample area unit. By measuring the fluoride in parts per million in a known volume of water, it was possible to calculate the total amount of fluoride ions released from the specimens. After each reading was taken, the total fluoride released in micrograms was calculated by multiplying the parts per million (1 ppm=1 $\mu\text{g}/\text{mL}$) by the water sample volume (10 mL). The total fluoride was then divided by the area of the sample disk to obtain the fluoride release in micrograms per square centimeter.^[10]

Statistical analysis

The data were analyzed with one-way analysis of variance (ANOVA) with a significance level of 0.05. Tukey *post-hoc* test was used to show groups with significant differences. Kruskal-Wallis test was used to compare the ARI scores. Data analyses were performed with the SPSS 15.0 software (Chicago, USA).

RESULTS

SBS and ARI

Descriptive statistics (means, median, standard deviations (SDs), maximum, and minimum values) for all groups are shown in Table 1 and Figure 2. The highest SBS was obtained for Etch + Breeze group (25.9 MPa), and the lowest value was recorded for G-CEM (3.5 MPa). The widest and shortest SDs was recorded for Etch + Breeze and G-CEM respectively (6.96, and 3.1 MPa).

One-way ANOVA showed significant differences with regards to SBS among the groups. The Tukey test comparisons indicated that mean SBS for Etch + Breeze was significantly higher than all other groups ($P<0.05$). The SBS of brackets cemented with Transbond XT were significantly higher than those of self-adhesives ($P<0.001$). However, when the self-adhesive resin cements were used with enamel etching, no significant differences was found in the SBS compared with Transbond XT, except for Breeze. After enamel etching, Breeze showed significantly higher SBS than Transbond XT ($P<0.001$).

After thermocycling, some brackets separated: 2 Brackets cemented with SET, 1 Breeze, 3 WetBond and 5 brackets cemented with G-CEM. The SBS for the failed brackets was considered as zero.

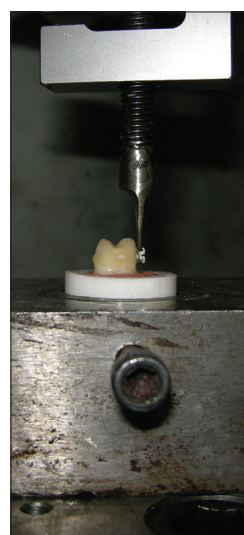


Figure 1: Position of the tooth and bracket in the Zwick testing machine, prior to the debonding process

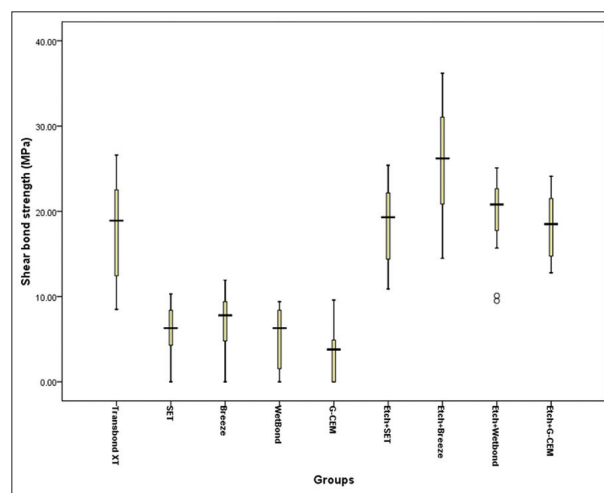


Figure 2: Box plot of the shear bond strength (SBS) test results megapascal. The bottom and top of the boxes are the 25th and 75th percentile (the lower and upper quartiles, respectively) of SBS for each group. The band near the middle of the box denotes the 50th percentile (the median) of SBS. The whiskers show the minimum and maximum of SBS in each group

Kruskal-Wallis test indicated that different adhesives had different bracket failure modes ($P < 0.001$) [Figure 3]. For SET, G-CEM and WetBond, all failures occurred at the enamel-adhesive surface. For Breeze, the majority of failures were at the enamel-adhesive surface. For Transbond XT, Etch + Breeze, Etch + SET, Etch + G-CEM and Etch + WetBond, most of the adhesive remained on the tooth (scores 2 and 3), indicating failure at the bracket-adhesive interface.

Fluoride release

Descriptive statistics (means, median, SDs, maximum, and minimum values) for fluoride release of all materials various days of the experiment has been summarized in Table 2 and Figure 4. Based on Kolmogorov-Smirnov test, data were normally distributed. Tukey test was used to investigate the differences between groups.

Day 1: Fluoride released by Fuji Ortho and Wetbond was statically higher than all other cements ($P < 0.05$), but there was no significant difference between these two ($P = 0.68$). G-CEM and Breeze were the second highest fluoride releasers. SET is released lower levels of fluoride compared to others in the first 24 h.

Day 2: Fluoride released by Fuji Ortho, Wetbond and Breeze was similar amongst them and statically higher than all other cements ($P < 0.05$). G-CEM was the second highest fluoride releaser. SET released statistically lower amounts of fluoride compared to all other cements ($P < 0.001$).

Day 3: Fluoride released by Fuji Ortho and Breeze was statically higher than all other cements ($P < 0.05$). WetBond, G-CEM and Breeze were ranked 2nd, 3rd, and 4th respectively.

Day 7: Fluoride released by Fuji Ortho and WetBond was statically higher than all other cements ($P < 0.05$). G-CEM and Breeze were the second highest fluoride releasers while SET released the lowest amounts of fluoride compared to all other cements.

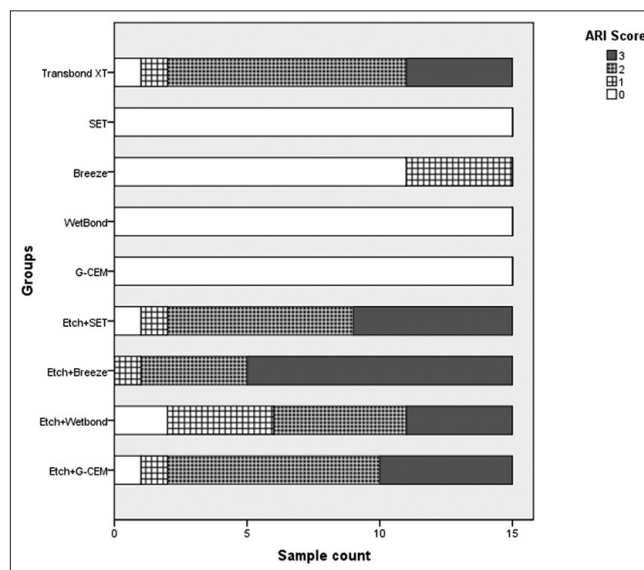


Figure 3: Stacked bars of the adhesive remnant index (ARI) scores. Each pattern denotes specific ARI score, ranging from 0 to 3

Table 1: Mean±SD and range for shear bond strength

| Experimental group (n=15) | Mean±SD | Min | Max |
|---------------------------|-------------------------|------|------|
| Transbond XT | 17.60±6.18 ^a | 8.5 | 26.6 |
| SET | 5.95±3.24 ^b | 0.0 | 10.3 |
| Breeze | 7.26±3.37 ^b | 0.0 | 11.9 |
| WetBond | 5.19±3.70 ^b | 0.0 | 9.4 |
| G-CEM | 3.53±3.09 ^b | 0.0 | 9.6 |
| Etch+SET | 18.42±4.93 ^a | 10.9 | 25.4 |
| Etch+Breeze | 25.86±6.96 ^c | 14.5 | 36.2 |
| Etch+WetBond | 19.47±4.75 ^a | 9.5 | 25.1 |
| Etch+G-CEM | 18.46±3.88 ^a | 12.8 | 24.1 |

^aThere is no significant difference between mean±SD of Transbond XT; Etch+SET; Etch+WetBond and Etch+G-CEM (P value>0.05), ^bThere is no significant difference between mean±SD of SET; Breeze, WetBond and G-CEM (P value>0.05), ^cStatistical significance level of Etch+Breeze; SBS–Shear bond strength; SD–Standard deviation

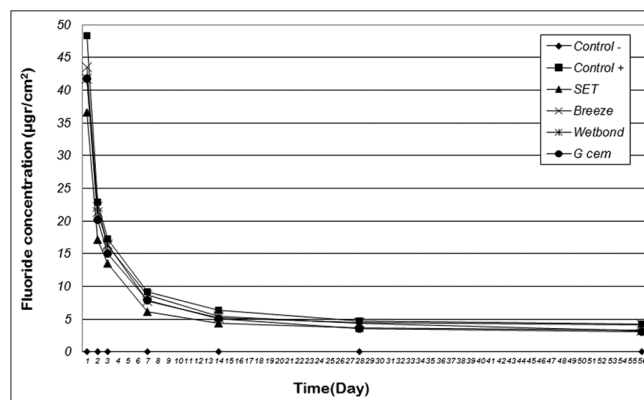


Figure 4: Mean fluoride release from the samples in micrograms per square centimeter

| Groups and time of measurement | Day 1 | Day 2 | Day 3 | Day 7 | Day 14 | Day 28 | Day 56 |
|--------------------------------|------------|------------|------------|-----------|-----------|-----------|-----------|
| Transbond XT (control -) | 0.00±0.00 | 0.00±0.00 | 0.00±0.00 | 0.00±0.00 | 0.00±0.00 | 0.00±0.00 | 0.00±0.00 |
| Fuji ortho (control +) | 48.35±2.11 | 22.87±1.06 | 17.3±0.71 | 9.17±0.31 | 6.37±0.31 | 4.75±0.19 | 4.25±0.19 |
| SET | 39.32±1.52 | 17.12±0.57 | 13.57±0.39 | 6.10±0.26 | 4.37±0.12 | 3.67±0.12 | 3.30±0.13 |
| Breeze | 43.55±2.00 | 21.32±1.39 | 16.43±0.62 | 7.78±0.38 | 5.13±0.12 | 4.47±0.22 | 4.17±0.10 |
| WetBond | 46.90±2.00 | 22.13±0.87 | 16.18±0.79 | 8.70±0.43 | 5.40±0.37 | 4.35±0.14 | 3.17±0.19 |
| G-CEM | 41.72±1.55 | 20.23±0.86 | 15.8±0.70 | 7.92±0.53 | 5.02±0.25 | 3.52±0.12 | 3.07±0.05 |

Day 14: Fluoride released by Fuji Ortho was statically higher than all other cements ($P < 0.05$) while on the second level, WetBond, Breeze and G-CEM released similar amounts of fluoride. SET released the lowest amounts of fluoride compared to all other cements.

Day 28: Fluoride released by Fuji Ortho was statically higher than all other cements ($P < 0.05$) while on the second level, WetBond, Breeze and G-CEM released similar amounts of fluoride. SET released the lowest amounts of fluoride compared to all other cements.

Day 56: Fluoride released by Fuji Ortho and Breeze was statically higher than all other cements ($P < 0.05$). SET and WetBond were the second highest fluoride releasers, while G-CEM was the third fluoride releaser.

DISCUSSION

Although the present investigation shows that the amount of fluoride released from the self-adhesive cements was generally lower compared to the glass ionomer, it should be pointed out that glass ionomer and WetBond released similar level of fluoride on days 1, 2, and 7. Breeze and glass released similar levels on days 3 and 56. From a clinical point of view, our results indicate that these cements release sufficient amounts of fluoride for an extended period of time. According to Rawls the fluoride released from the self-adhesive cements in the current study is even higher than the level required to prevent the demineralization of the enamel.^[11]

There is no standard method to test SBS in orthodontics. Our methodology design is based on the study by Al-Saleh and El-Mowafy.^[2] Our final results with regards to Transbond XT and Breeze are in accordance with Al-Saleh and El-Mowafy study.^[2] The SBS of the self-adhesive cements was low and 11 teeth were debonded before reaching the loading cycle. Although there is no consensus on what range to consider as the acceptable SBS for cements used in orthodontics,^[12-16] some studies suggest a range of 6.5-10 MPa as the acceptable range.^[13,14] Some authors mention that a minimum of 8 MPa is required in order to retain the bracket on the tooth.^[15,16]

Our results show that Breeze cement had the highest SBS compared to the other self-adhesive groups, with a mean SBS of 7.26 MPa. All other self-adhesive cements, which were applied on non-etched surfaces, had lower mean SBS than the threshold as suggested by the literature. Thus, the application of these cements on non-etched surfaces is not recommended in a clinical setting. However, when these cements were applied on etched surfaces, they showed consistently higher SBS, ranging from 10.9 MPa to 36.2 MPa.

According to Table 1, the widest range of SD for mean SBS was recorded for Transbond XT and Etch + Breeze groups. In the Etch + Breeze group, the wide SD can be attributed to the high mean SBS. However, for the Transbond XT group, this wide SD was more likely due to the fact that the cement did not consistently create strong bonds with all the teeth. This finding is particularly important in the clinical setting, since it indicates that this cement might not perform predictably in the clinic. From this perspective, Etch + WetBond, Etch + G-CEM and Etch + SET are considered to be clinically superior compared to Transbond XT due to the relatively narrower range of SD around their mean SBS. The three mentioned groups can also be considered safer than the Etch + Breeze, since their comparatively lower SBS (compared to the unusually high Breeze SBS) leads to decreased risk of enamel micro-fractures. However, due to the pattern of bonding failure in the Etch + Breeze, which occurs in the bracket-cement interface, the risk of micro-fracture is less likely.

According to our study, the pattern of failure was similar among self-adhesive resins and mainly occurred at the enamel-adhesive surface. On the other hand, most of failures in the etched groups occurred between the bracket and adhesive. The latter was observed in the Transbond XT group as well. The Etch + Breeze group had the highest ARI score and thus has the strongest adhesion to tooth enamel. This high ARI score indicates the low possibility of enamel micro-fractures due to the application of this cement. This is important since the site of failure provides useful clinical information about the bond.^[2] A bond that fails at the interface of the enamel and cement is favorable, since it makes cleaning and polishing of the teeth easier, resulting in reduced enamel loss during the cleaning procedures to remove adhesive remnants. However, this failure increases the chances of enamel damage.^[17]

When teeth were acid-etched prior to the application of cements, bonding failures were observed at the bracket-cement interface. Some authors have considered the bond failure at the bracket-adhesive interface to be safer than fracture at the enamel-adhesive interface, due to the decreased chances of enamel micro-fracture. However, this pattern results in more residual adhesive on the tooth after debonding, increasing the chair time spent on the removal of the residual adhesive.^[18]

All in all, since the self-adhesive cements in this study showed sufficient bonding on etched-surfaces, their clinical application in bonding of brackets to enamel surfaces is recommended. Clinicians might also benefit from the favorable working time, simple manipulation, auto-mixing and lower cost of these cements as well as the release of fluoride and elimination of priming procedure.

CONCLUSION

Considering the limitations of lab studies, it can be concluded that:

- The SBS of the self-adhesive systems used for bonding orthodontic brackets was significantly lower than Transbond XT. Thus, the application of these cements on non-etched surfaces is not recommended in a clinical setting
- Bracket failure modes were different between the etched and non-etched surfaces, with the majority of bracket-cements failures on the etched surfaces and enamel-cement failures on the non-etched surfaces
- All self-adhesive cements released clinically sufficient amounts of fluoride for an extended period of time, which can prevent the demineralization of the enamel.

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