

Orthodontic bonding with self-etching primer and self-adhesive systems

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SUMMARY The purpose of this study was to compare the tensile bond strengths of orthodontic brackets bonded to enamel using conventional multi-step adhesive, self-etching primer (SEP), which combines etching and priming into a single step, and self-adhesive systems, which combine etchant, primer, and adhesive. Metal brackets were bonded to 90 extracted human premolars according to three experimental protocols: group 1, conventional multi-step adhesive system; group 2, SEP; and group 3, self-adhesive system. All specimens were debonded using an Instron universal machine and failures between the tooth surface and bracket base were observed by scanning electron microscopy (SEM). The bracket bases were then analysed by mapping of energy-dispersive X-ray (EDX) spectrometry to calculate the distributive percentages of enamel or resin. The bond strength, percentage distribution, and calcium on the debonded interface were determined and analysed by one-way analysis of variance, and means were ranked by a Tukey interval, calculated at the 95 per cent confidence level.

Group 1 produced the greatest bond strength, followed by groups 2 and 3. Group 3 showed the highest debonded interface between resin and enamel or within the resin itself, followed by groups 2 and 1. Groups 1 and 2 displayed significantly more debond failures at the interface between the bracket and the resin than group 3. More calcium particles were observed on the bracket base after debonding in group 3 than in groups 2 and 1. The simplified bonding procedures caused an undesirable decrease in tensile bond strength.

Introduction

To minimize the number of steps when bonding and to reduce clinical chair time, self-etching primers (SEPs) and self-adhesive systems were developed. SEPs combine conditioning and priming into a single treatment step (Cinader, 2001; Miller, 2001), which does not require acid etching. The self-adhesive system not only has acidic monomers that demineralize and infiltrate the tooth substrate, resulting in micromechanical retention, but it is also capable of forming chemical adhesion with enamel, metals, and composite materials. Therefore, SEPs do not require acid etching and self-adhesives need neither prior acid etching nor priming (De Munck *et al.*, 2004; Vicente *et al.*, 2005; Gerth *et al.*, 2006; Basaran *et al.*, 2009). The active ingredient for both the SEPs and the self-adhesive systems is methacrylated phosphoric acid ester, which etches the enamel surface and primes at the same time. The phosphate group of the methacrylated phosphoric acid ester dissolves calcium and removes it from hydroxyapatite (Miller, 2001). However, rather than being rinsed away, the calcium forms a complex with the phosphate group and becomes incorporated into the network when the primer polymerizes (Cinader, 2001).

These products save time and require less effort in clinical practice. However, there is always the concern as to whether they achieve the same level of bond strength as conventional products.

In orthodontic bond strength studies, analysis of variance (ANOVA) has commonly been used to compare the results of three or more groups. However, abnormal distribution of the data is likely to be found in sample sizes of less than 10 specimens per group. It has been suggested that at least 20, and preferably 30, specimens per experimental group should be utilized (Fox *et al.*, 1994).

A number of *in vitro* experiments (Vicente *et al.*, 2005; Bishara *et al.*, 2006; Sethusa *et al.*, 2009) have compared their results with the findings of Reynolds (1975). However, the clinically acceptable bond strength proposed by that author was tensile bond strength, and shear and tensile strengths show different results even using the same material (Ostertag *et al.*, 1991; Bhatt *et al.*, 1996; Jobalia *et al.*, 1997; Katona and Long, 2006).

Most of the above studies failed to address the location at which bond failure occurred. Furthermore, the mechanism of mechanical retention on the etched enamel had to be rinsed off from the surface of the enamel rods with an

abundant water spray before bonding. The etched enamel with microspores forms resin tags with the orthodontic resin to produce the bonding (Zachrisson and Büyükyılmaz, 2005). If the releasing calcium cannot be rinsed off from the microspores on etched enamel, it may affect microretention and cause a reduction in bond strength. Hence, the purposes of this investigation were three-fold:

1. To compare the tensile bond strength of orthodontic brackets bonded with conventional multi-step adhesive, SEP and self-adhesive systems.
2. To examine the debonded interface on the bracket base with a scanning electron microscope (SEM) and energy-dispersive X-ray spectrometry (EDX).
3. To detect the calcium remaining on the bracket base after debonding.

Materials and methods

Ninety premolars extracted for orthodontic purposes from 9- to 16-year-old subjects were used following informed consent. The 90 teeth were divided into three equal groups, washed under tap water, and stored in a closed plastic box containing a physiologic saline solution. The teeth were tested within 3 months of extraction (Jameson *et al.*, 1994). The criteria for tooth selection were as follows: (1) the crown was perfect with no defect and (2) the tooth had never been pre-treated with a chemical agent, such as hydrogen peroxide or formalin. The contour of the labial surface of the crown was adapted to the base of the bracket before bonding. Ninety mini-Dynalock upper premolar brackets (118-503, 3M Unitek, Monrovia, California, USA) were used. The bracket base was formed of mesh-shaped arc with a surface area of approximately 3.1×3.4 mm (10.54 mm^2), which could be easily fitted onto the curvature of the buccal surface of the premolar. The buccal surface of each crown was polished with pumice powder (Prophyol fine particle, Myco Industries, Philadelphia, Pennsylvania, USA) paste containing no fluoride or oil for 10 seconds and subsequently rinsed with an abundant water spray for 10 seconds, followed by drying with an air spray. The outline of the bracket base was demarcated on the etched buccal enamel with a pencil. The surface outside the demarcated area was coated with nail varnish before bonding to standardize the bonding area. The brackets were then bonded onto the buccal surfaces of the premolars according to the manufacturer's instructions.

Group 1: Conventional bonding system. A 15 per cent H_3PO_4 solution (Wang *et al.*, 1994) was applied to the enamel surface for 15 seconds (Wang and Lu, 1991). Subsequently, a layer of Transbond XT (3M Unitek) primer was applied to the etched tooth and Transbond XT paste to the base of the bracket. The bracket was then pressed firmly to the tooth. Excess adhesive was removed with an explorer. The adhesive was light cured using a halogen lamp (Curing

light 2500, 3M Corp, St Paul, Minnesota, USA) positioned approximately 1 mm from the top of the bracket for 40 seconds (Wang and Meng, 1992).

Group 2: SEP system. A layer of Transbond Plus (3M Unitek) primer was rubbed on the tooth for 3 seconds and Transbond XT paste was applied to the base of the bracket. The bracket was then pressed firmly onto the tooth and light cured for 40 seconds.

Group 3: Self-adhesive system. The RelyX Unicem Aplicap (3M Espe) was activated in the Aplicap Activator (3M Espe AG Dental Products) after which the capsule was mixed for 10 seconds in a high-frequency mixing unit (Rotomix, 3M Espe). The capsule was then inserted in the Aplicap Applier (3M Espe) and the cement was applied to the base of the bracket. The bracket was pressed firmly onto the tooth and the adhesive was light cured for 40 seconds.

The specimens were embedded in a dental hard stone contained in a plastic cup with the buccal surface and bracket exposed. After the stone had set, all specimens were immersed in distilled water at 37°C for 24 hours.

Tensile bond strength was measured with an Instron universal machine (AGS-1000 kGW, Autograph, Shimadzu Corp., Chiroda-Ku, Tokyo, Japan) connected to a 50 N load cell at a crosshead speed of 2 mm/minute (Klocke and Kahl-Nieke, 2005). The force required to debond each bracket was registered in newtons and then converted into megapascals (MPa) as a ratio of newtons to surface area of the bracket.

The modified adhesive remnant index (ARI; Årtun and Bergland, 1984) used to assess the debonded interfaces of the bracket base and the enamel surface was examined using a SEM (Jeol JSM 6400, Cambridge, UK) under $\times 20$ magnification. EDX (LinkISIS 300, Oxford, UK) was used to detect the different chemical elements on the debonded interfaces. The bracket bases were then mapped and the percentages of the debonded interface mapping area were calculated with the soft imaging system software (Soft

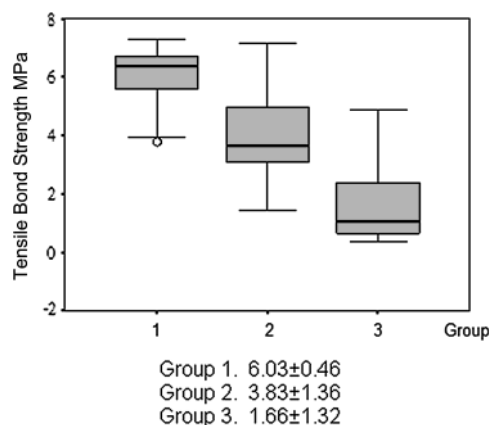


Figure 1 Tensile bonding strength achieved with the three systems. Group 1, conventional system; group 2, self-etching primer system; and group 3, self-adhesive system.

imaging system GmbH 2000, Soft Imaging System Corp., Lakewood, Colorado, USA). These procedures have been described previously (Wang and Lu, 1991).

The bond strength and distributive percentage of the debonded interface were determined and analysed with the Statistical Package for Social Sciences (SPSS Inc., Chicago, Illinois, USA) by one-way ANOVA and means were ranked by a Tukey interval, calculated at the 95 per cent confidence level (Grafen and Hails, 2002).

Results

The mean and standard deviations of bond strengths are given in Figure 1. The statistical analysis of bond strength with one-way ANOVA gave an F value of 89.7, which showed a statistically significant difference ($P < 0.01$). Tukey test ($\alpha = 0.05$) was chosen for further analysis and comparison and revealed statistically significant differences ($P < 0.01$). Group 1 showed a significantly higher bond strength, followed by group 2. The lowest tensile bond strength was found in group 3.

The distributive percentages of the various debonded interfaces in each group are given in Table 1. Statistical analysis of the debonded interface with one-way ANOVA gave an F value of 21.3, which was statistically significantly different ($P < 0.05$). Tukey test ($\alpha = 0.05$) was chosen for further analysis and comparison and revealed statistically significant differences ($P < 0.05$). Group 3 showed a

Table 1 Mean and standard deviation (SD) of the debond interface distributions of the three groups (percentage).

Debond interface	Bracket–resin		Enamel–resin or within the resin itself		Calcium particle	
	Mean	SD	Mean	SD	Mean	SD
Group						
1. Conventional multi-step adhesive	62.43*	14.61	31.73	11.60	5.50	5.04
2. Self-etching primer system	54.20*	23.12	37.63	13.72	8.10	9.49
3. Self-adhesive system	34.77	22.53	51.67*	16.66	14.03*	9.82

* $P < 0.05$.

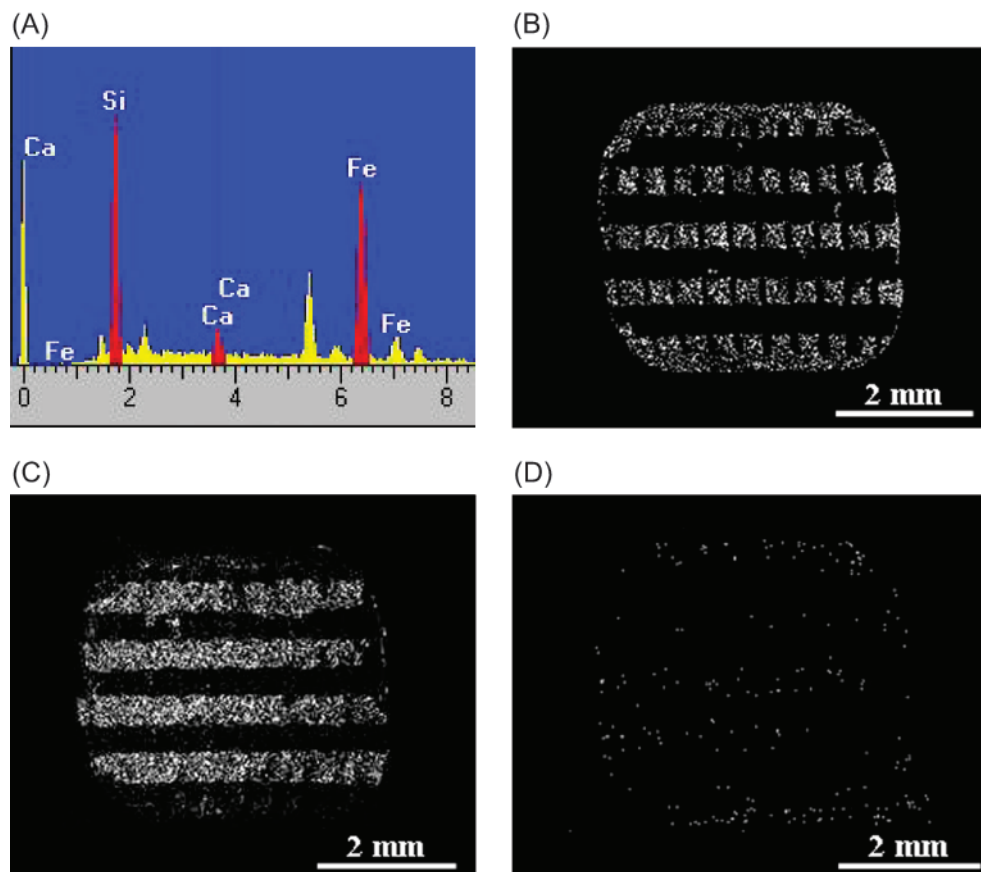


Figure 2 Bracket debond interface mapping of the conventional acid-etching group, scanning electron microscopy $\times 20$ magnification. (A) The energy-dispersive X-ray spectrum on the bracket base composed of iron, silicon, and calcium. (B) Iron map. White spots indicate detected iron and represent the debonded area between the bracket and the resin. (C) Silicon map. White spots indicate detected silicon and represent the debonded area between the enamel and the resin or within the resin itself. (D) Calcium map. Diffuse white spots indicate calcium particles.

significantly highest debonded interface either between the resin and enamel or within the resin itself, followed by groups 2 and 1. Groups 1 and 2 displayed significantly more debond failures at the interface between the bracket and the resin than group 3.

A significantly higher percentage of iron was found in groups 1 (Figure 2) and 2 indicating more debond failure at the bracket–resin interface (Figure 3). In contrast, in group 3, failure was greater at the resin–enamel interface or within

the resin (Figure 4). Significantly higher percentages of calcium were found in group 3 than in groups 1 and 2 (Figure 5).

Discussion

The findings of this research show that the conventional multi-step adhesive system resulted in the greatest bond strength, followed by the SEP system. The lowest bond

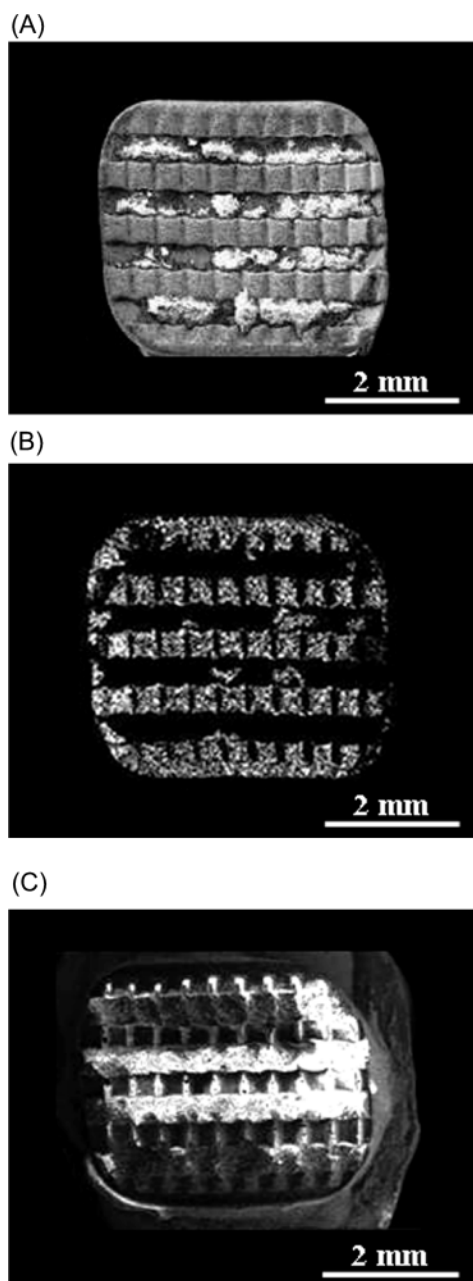


Figure 3 The debonded bracket interface of the self-etching primer system. (A) Scanning electron microscopy (SEM) image of the bracket base. (B) Iron map. White spots indicate Fe mapping area of the bracket base, which represents debond failure at the bracket–resin interface. (C) SEM image of the tooth interface. $\times 20$ magnification.

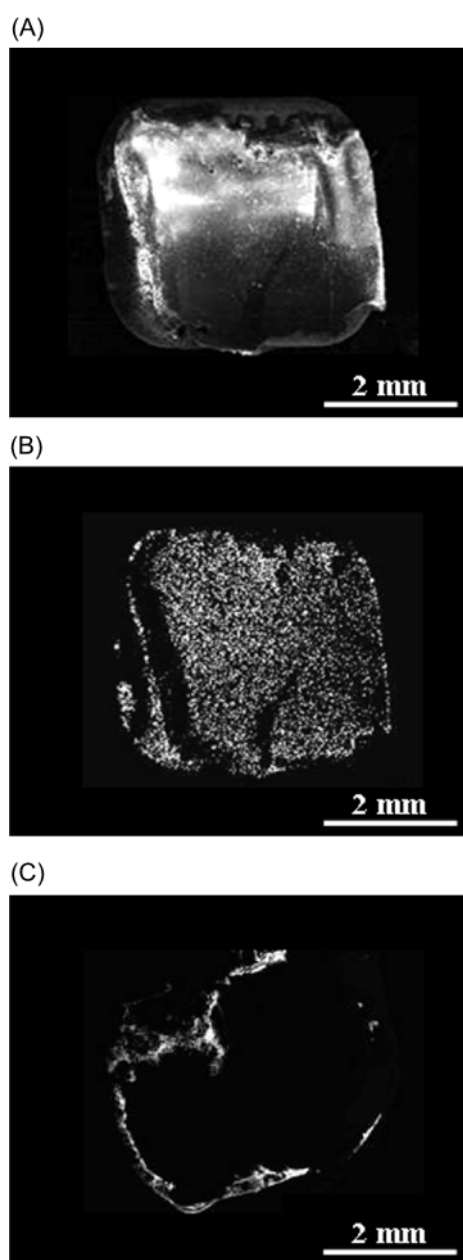


Figure 4 The debonded interface of a bracket base in the self-adhesive group. (A) Scanning electron microscopy (SEM) image of bracket base. (B) Silicon map. White spots indicate Si, which is found in the bracket interface. (C) SEM image of the enamel. This indicates debonded failure at the resin–enamel interface or within the resin itself. $\times 20$ magnification.

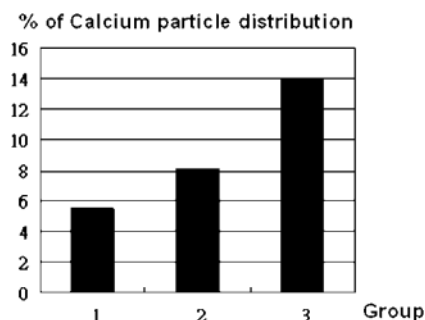


Figure 5 Calcium particle distribution percentage on the debonded bracket interface. Group 1, conventional system; group 2, self-etching primer system; and group 3, self-adhesive system.

strength was achieved with the self-adhesive system. This indicates that the simplified bonding procedures caused an undesirable decrease in tensile bond strength.

The results showed a remarkably low tensile bond strength of the self-adhesive system, which might be due to the different loading mode. Katona and Long (2006) indicated that tensile bond strength of the single adhesive is much lower than shear bond strength. This could be attributed to the 'load' and 'structural factors' and the ability (strengths) of the structural constituents to withstand such stresses.

According to the often-cited values given by Reynolds (1975), the minimum clinically acceptable tensile bond strength is approximately 5 MPa. Therefore, the tensile bond strength of Transbond Plus and RelyX Unicem in this study may not be sufficient for orthodontic bonding. Consideration should be given to increasing the application time. One explanation suggested in previous studies (Bishara *et al.*, 1998; Cal-Neto and Miguel, 2006) is that the short application time results in poor quality etching. Bishara *et al.* (2006) indicated that the shear bond strength of Transbond Plus (5.9 ± 2.7 MPa) is sufficient when used to bond orthodontic brackets. In their study, the SEP was applied to the enamel of human molars for 15 seconds prior to bracket bonding, which differs from the manufacturer's suggested time.

RelyX Unicem has the same active ingredient as SEP. However, the active ingredient of the self-adhesive resin has to react with its acid. Its acidity not only dissolves the enamel calcium but also is neutralized with alkaline filler particles to create chemical adhesion with the enamel (Abo-Hamar *et al.*, 2005). It is also noteworthy that the pH value of RelyX Unicem was 2.8 after mixing for 3 minutes and apparently increased to 5.0 at 20 seconds after light curing (Han *et al.*, 2007). The acidity drops significantly after light curing, which leads to less penetration of the adhesive through the enamel, causing poor bond strength.

When assessing the site of debond failure, most studies (Arnold *et al.*, 2002; Büyükyilmaz *et al.*, 2003; Grubisa *et al.*, 2004; Rajagopal *et al.*, 2004; Trites *et al.*, 2004) used the ARI. However, the ARI is largely subjective and it is

difficult to discriminate between tooth and resin on the debonded surface (Årtun and Bergland, 1984). In the present study, the debonded surfaces of the bracket bases were examined with SEM and EDX to analyse the distributive percentages because EDX is sensitive to atom distribution.

Groups 1 and 2 displayed significantly more debond failure at the interface between the bracket and the resin than group 3, perhaps indicating that in both groups 1 and 2 there was a strong bond between the tooth and the resin. Less debond failure was observed for the self-adhesive at the interface between the bracket and the resin and less adhesive remained on the tooth surface after debonding.

No enamel detachment occurred in this study. Further analysis is still needed to evaluate if there is any change in the bond failure pattern and enamel detachment of these adhesive systems after tensile bond strength is increased.

Conclusion

The simplified bonding procedures caused an undesirable decrease in tensile bond strength. The conventional multi-step adhesive system is still supported.

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