Inadequate shear bond strengths of self-etch, self-adhesive systems for secure orthodontic bonding

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This study evaluated the shear bond strength (SBS) of a traditional orthodontic bracket bonding agent (Transbond XT) against two self-etch, self-adhesive systems (Maxcem Elite and Vertise Flow). Sixty premolar teeth and sixty brackets were randomly and equally divided into five groups: Transbond XT (TXT) as the control, Maxcem Elite (ME) without etching, ME with etching (ME/Etch), Vertise Flow (VF) without etching, and VF with etching (VF/Etch). Respective SBS results of the five groups were 9.86±3.20, 4.67±2.94, 7.82±2.56, 2.55±0.77, and 7.89±1.17 MPa. SBS values of the new self-etch adhesive systems were significantly lower than the traditional etch-and-rinse control ($p<0.005$). However, no significant differences were found between TXT and the self-adhesives applied with etching ($p>0.005$). After debonding, ARI scores “0” and “1” were predominant in non-etched ME and VF groups. It was concluded that new self-etch, self-adhesive bonding systems require additional phosphoric acid application to achieve comparable SBS values as the traditional orthodontic bonding agent.

Keywords: Shear bond strength, Self-adhesive resin cement, Self-adhesive flowable composite, Orthodontic bracket bonding

INTRODUCTION

Since Buonocore introduced the technique of acid-etching enamel surfaces in 1955[1], adhesive dentistry has revolutionized the way dentistry is practiced today[2-5]. Dental adhesive systems are embraced and employed by dentists worldwide, and they have become indispensable in the bonding of orthodontic brackets to enamel. A perennial challenge and unrelenting focus in the research and development of orthodontic adhesive systems is to deliver strong and reliable bonding for bracket placement on the one hand, and to maintain a sound, unblemished enamel surface after debonding of orthodontic brackets on the other hand[2,6].

In this age of adhesive dentistry, tooth-conserving and time-saving adhesive methods of retaining orthodontic attachments have replaced traditional methods and procedures. Although three-step etch-and-rinse systems were hailed as a major breakthrough in adhesive dentistry, the pursuit for simpler and easier adhesive systems has led to these developments: two-step etch-and-rinse systems (TSERs) which combine priming and bonding functions in a single bottle; two-step self-etch systems (TSSEs) which combine conditioning and priming functions in a single bottle; and all-in-one adhesive systems (AASs) which combine all the three functions in a single bottle[7,8]. Apart from etch-and-rinse and self-etch approaches, resin-modified glass ionomer cements (RMGIC) are another alternative for bonding orthodontic brackets[9].

A common goal of contemporary adhesive system manufacturers is to design and develop bonding agents that offer the fastest, easiest and best quality of bonding. However, with any kind of simplification in clinical application procedure, there arises a concern in loss of bonding[7]. During the last decade, numerous studies were carried out to investigate the efficiency, durability, and robustness of contemporary bonding agents by evaluating their shear bond strength or tensile bond strength[6,10-15]. While most TSERs[11], TSSEs[11], and AASs[12] were found to be suitable for orthodontic use, flowable composites failed to provide sufficient bonding strength for bracket attachment[12,16].

Flowable composites were first introduced in 1995 to restore Class V lesions[17]. They reportedly have excellent handling properties, low viscosity, and superior injectability with non-stickness[18]. Easy handling is a highly desired characteristic because it reduces the working time of clinicians and chairside time of patients[19]. Various studies had shown that flowable composites exhibited comparable shear bond strengths as orthodontic adhesive Transbond XT and were promising materials for orthodontic bracket bonding[12,20,21].

Vertise Flow (Kerr, Orange, CA, USA) is a recently launched, self-adhesive flowable resin composite. It is marketed as an adhesive-free restorative material

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indicated for the restoration of small Class I and V cavities and non-carious cervical lesions, for repairing porcelain chips and defects, as a liner for large Class I and II restorations, or as a pit and fissure sealant[22]. As the easy handling of Vertise Flow makes it an attractive candidate for orthodontic bracket bonding, its water sorption property was recently evaluated *in vitro*[23,24].

To simplify clinical procedures and replace technique-sensitive, multi-step adhesive systems[25], self-adhesive resin cements were introduced in 2002. They can be directly applied on the dentin surface without any substrate pretreatment for luting. Chemical bonding to the tooth substrate is achieved by a reaction between phosphoric acid monomers and hydroxyapatite present in dental hard tissues[26]. However, numerous studies have reported that self-adhesive cements exhibited lower bond strengths than conventional resin cements that rely on the use of etch-and-rinse adhesive systems[27-30].

The aim of this study was to evaluate the shear bond strengths (SBSs) of two self-etch, self-adhesive resin materials with and without phosphoric acid etching and compare them against a traditional orthodontic adhesive. The null hypotheses of this study were:

1. There would be no statistically significant differences in SBS between a traditional orthodontic adhesive and self-etch, self-adhesive resin materials with phosphoric acid etching.
2. There would be no statistically significant differences in SBS among the self-etch, self-adhesive resin materials with or without phosphoric acid etching.

MATERIALS AND METHODS

Tooth specimens

Sixty upper first premolar teeth extracted for orthodontic purposes were used in this study. Teeth with enamel hypoplasia, fractures, or caries were excluded. After eliminating all soft tissue remnants and other extraneous material, the teeth were cleaned with pumice for 15 s. Each tooth was then rinsed with tap water for 15 s. All teeth were mounted vertically in self-cure acrylic resin blocks with their labial surfaces exposed.

Experimental groups

The teeth were randomly divided into five groups containing 12 teeth per group. Compositions and details of the three adhesive materials used in this study are shown in Table 1. Metal brackets (Master Series, American Orthodontics, Sheboygan, WI, USA) for upper first premolars were bonded to the teeth according to each adhesive system manufacturer's instructions as follows:

1. Transbond XT (TXT) control group: Enamel surfaces were etched for 15 s using 37% phosphoric acid (Gel Etch, 3M Unitek, Monrovia, CA, USA), rinsed with water for 15 s, and air-dried for 15 s. After Transbond XT primer (3M Unitek, Monrovia, CA, USA) was applied to the enamel surfaces, a bracket base (10.27 mm^2 surface area according to manufacturer) was bonded in place on each etched enamel surface using Transbond XT adhesive. Traditional orthodontic adhesive was polymerized using a light-emitting diode (LED) curing light (Demi, Kerr, Orange, CA, USA) for 20 s each on both mesial and distal sides (*i.e.*, a total of 40 s).
2. Maxcem Elite (ME) group: Maxcem Elite was applied on enamel surfaces, and bracket bases were bonded in place without separate enamel etching step before bonding. This self-etch, self-adhesive resin cement was polymerized with LED curing light for 20 s each on both mesial and distal sides (*i.e.*, 40 s total).

Table 1 Compositions of adhesive materials used in this study

Adhesive material	Manufacturer	Batch No.	Filler weight (%)	Composition
Transbond XT Light-cure adhesive	3M Unitek, Monrovia, CA, USA	Ethctant: 110214 Adhesive: N180717 CX5PN	80	35% phosphoric acid, silane-treated quartz, bisphenol A diglycidyl ether dimethacrylate, bisphenol A bis-(2-hydroxyethyl)-ether dimethacrylate, silane-treated silica
Vertise Flow Self-adhesive	Kerr Corporation, Orange, CA, USA	3444010	70	GPDM, HEMA, MEHQ, prepolymerized particles, Ba glass, colloidal SiO_2, YbF_3, ZnO, 1 μm for Ba glass; nanoscale SiO2 and YbF_3. Overall mean: 1 μm Filler loading: not specified
Maxcem Elite Self-etch, self-adhesive	Kerr Italia, 84018 Scafati, Italy	3606187	69	GPDM, methacrylate ester monomers, HEMA, 4 methoxyphenol, cumene hydroperoxide, titanium dioxide and pigments

GPDM: Glyceroldimethacrylate dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; MEHQ: Monomethyl ether hydroquinone

3. ME with Etching (ME/Etch) group: Enamel surfaces were etched for 15 s using 37% phosphoric acid (Gel Etch, 3M Unitek, Monrovia, CA, USA), rinsed with water for 15 s, and air-dried for 15 s. ME resin cement was polymerized with LED curing light for 20 s each on both mesial and distal sides (*i.e.*, 40 s total).
4. Vertise Flow (VF) group: Vertise Flow was applied on enamel surfaces, and bracket bases were bonded in place without separate enamel etching step before bonding. This flowable composite was light-cured using LED for 20 s each on both mesial and distal sides (*i.e.*, 40 s total).
5. VF with Etching (VF/Etch) group: Enamel etching procedure was as per that for ME/Etch group. After VF was applied on enamel surfaces and bracket bases were bonded in place, flowable composite was light-cured using LED for 20 s each on both mesial and distal sides (*i.e.*, 40 s total).

Shear bond strength test

After bracket bonding procedure, all specimens were stored in distilled water at 37°C for 24 h. Shear bond strength (SBS) of each group was measured using a universal testing machine (AGS-X, Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min. Shear force was applied parallel to the long axis of each tooth. The force required to shear off the bracket was directly recorded in Newtons (N) and converted into megapascal (MPa) using the following equation:

Shear force (MPa)=Debonding force (N)/Bracket surface area (mm^2) where 1 MPa=1 N/mm^2 and bracket surface area is 10.27 mm^2.

Recording of bracket bonding procedure duration

Duration of the entire bracket bonding procedure was recorded using a chronometer. Individual recordings were made for the duration of each step, from the beginning of tooth specimen's preparation to the end of light-curing the adhesive material.

For Transbond XT group, these steps were carried out: tooth scaling and polishing, drying, etching, rinsing, drying, priming, bracket placement, and light-curing of adhesive. For Maxcem Elite and Vertise Flow groups without etching, the following steps were omitted: etching, rinsing, drying, and priming. For Maxcem Elite and Vertise Flow groups with etching, only the priming step was omitted.

Adhesive remnant index (ARI) measurement

Enamel surfaces of bracket-bonded specimens were examined under an optical microscope (Leica Microsystems, Germany) at ×16 magnification to determine the amount of residual adhesive remaining on each tooth. Adhesive remnant index (ARI) scores[31,32] ranging from 0 to 3 were given as follows —0: No adhesive remained on tooth; 1: Less than 50% of adhesive remained on tooth; 2: More than 50% of adhesive remained on tooth; 3: All the adhesive remained on tooth.

Statistical analysis

For SBS, descriptive statistics including the mean, standard deviation, and minimum and maximum values were calculated for each group. SBS data were evaluated statistically among the groups using Kruskal-Wallis test. Pairwise comparisons were performed using Mann-Whitney U test with Bonferroni correction (number of comparisons=10). Level of significance was set at p=0.005.

Relationship between SBS and bracket bonding procedure duration of the adhesive materials was evaluated using correlational analysis. A chi square test was used to evaluate the differences in ARI scores among all the test groups. Data were analyzed using SPSS 13 for Windows software (SPSS Inc., Chicago, IL, USA).

RESULTS

Shear bond strength

Mean SBS values (in MPa) of the five test groups and their descriptive statistics are shown in Table 2 and Fig. 1 respectively. Transbond XT exhibited the highest SBS (9.86±3.20 MPa), and there were no statistically significant differences among Transbond XT, VE/Etch (7.89±1.17 MPa), and ME/Etch (7.82±2.56 MPa) groups (p>0.005). However, there were significant differences in SBS among Transbond XT, VE, and ME (p<0.005).

Among the five test groups, VF exhibited the lowest SBS (2.55±0.77 MPa) followed by ME (4.67±2.94 MPa). There were no significant differences in SBS between VF and ME (p>0.005). However, statistically significant differences were found between ME/Etch and VF as well as between VF/Etch and VF (p<0.005).

Relation between SBS and bracket bonding procedure duration

Figure 2 shows the correlational analysis outcome between SBS data and bracket bonding procedure durations. Positive strong correlation was found between SBS and bonding procedure duration, indicating that quicker bonding procedures resulted in low bonding strengths (r=0.885, p=0.046). When etching step was omitted, the obtained SBS values were not adequate for secure bracket bonding. However, SBS increased significantly (p<0.005) when teeth were pretreated by etching.

ARI scores

Table 3 shows the ARI scores of each test group. Figure 3 shows the distributions of ARI scores, by percentage, for all the test groups. Chi square test showed that there were statistically significant differences among the groups (p<0.001).

DISCUSSION

This *in vitro* study compared the shear bond strengths of different adhesive systems when they were used to bond metallic orthodontic brackets. Results of this study supported null hypothesis (1) that there were no statistically significant differences in SBS between traditional orthodontic adhesive (TXT) and self-etch, self-adhesive resin materials with phosphoric acid etching (ME/Etch and VF/Etch). However, null hypothesis (2) was partially rejected. On the one hand, there were no statistically significant differences in SBS among self-etch, self-adhesive resin materials with and without phosphoric acid etching. On the other hand, statistically significant differences were found between ME/Etch and VF as well as between VF/Etch and VF ($p<0.005$).

Transbond XT

Minimum bond strength for clinically acceptable bracket bonding was reportedly 6–8 MPa[33]. In the present study, Transbond XT and self-etch, self-adhesive resin materials with phosphoric acid etching showed clinically acceptable bond strengths for bracket bonding. Transbond XT is a traditional composite resin filled with filler particles of diverse sizes[34] and which reportedly exhibited clinically acceptable bond strength values[34,35].

Transbond XT was used as the gold standard in many SBS studies[14,31]. However, its SBS values varied widely between 8.9±3.9 and 18.1±5.5 MPa in literature[35,36]. Mineral structures of the teeth used in these studies might have an effect on the SBS of different bonding agents, thus accounting for this large disparity in the SBS data of Transbond XT. Secondly, pressure exerted by the handpiece instrument during polishing might have an effect on tooth surface features, which would then affect the SBS of adhesive materials and cause differences in the SBS of Transbond XT among different studies. These conjectures and considerations bear significant clinical impact and should be proved

Table 2 Mean SBS values and their standard deviations

Group	SBS value (MPa)
Transbond XT (TXT)	9.86 (3.20)^{A}
Maxcem Elite (ME)	4.67 (2.94)^{BC}
Maxcem Elite with Etching (ME/Etch)	7.82 (2.56)^{AB}
Vertise Flow (VF)	2.55 (0.77)^{C}
Vertise Flow with Etching (VF/Etch)	7.89 (1.17)^{AB}

Different capital letter superscripts indicate that SBS values are significantly different at $p<0.005$.

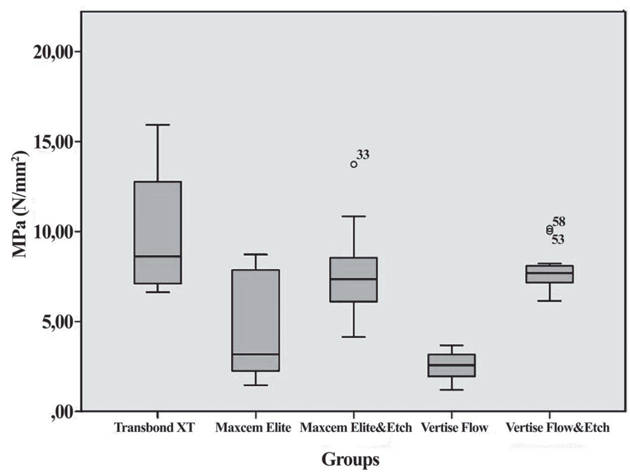

Fig. 1 Descriptive statistics of SBS (MPa) of test groups in this study.

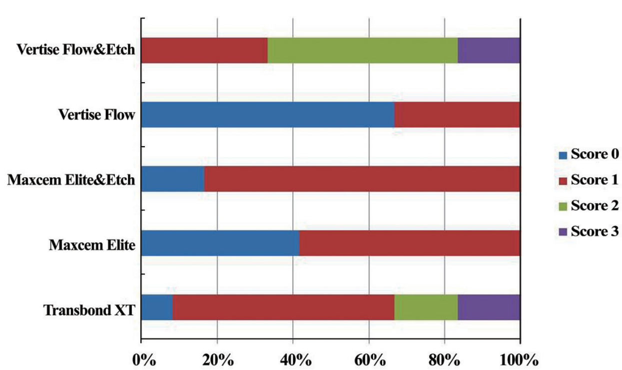

Fig. 2 Correlation between SBS values and bracket bonding procedure durations.

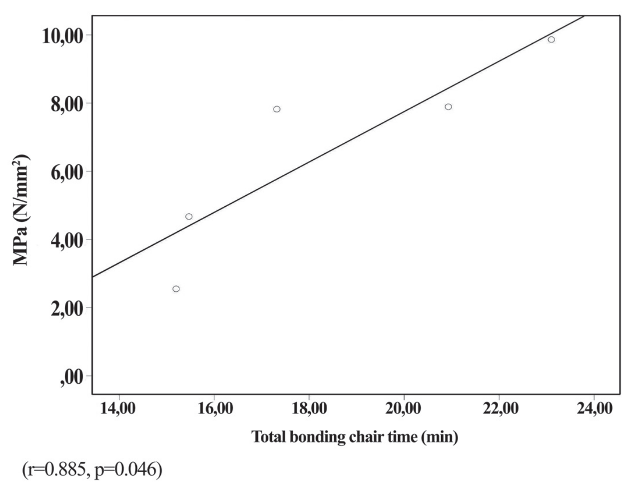

Fig. 3 Distribution chart of ARI scores in test groups.

Table 3 Distribution of ARI scores in each test group

Group	Score 0 (%)	Score 1 (%)	Score 2 (%)	Score 3 (%)	Total (%)
TXT	1(8)	7(58)	2(17)	2(17)	12(100)
ME	5(42)	7(58)	0(0)	0(0)	12(100)
ME/Etch	2(17)	10(83)	0(0)	0(0)	12(100)
VF	8(67)	4(33)	0(0)	0(0)	12(100)
VF/Etch	0(0)	4(33)	6(50)	2(17)	12(100)

Statistically significant differences in ARI score among the five test groups (p<0.001).
0: No adhesive remained on tooth; 1: Less than 50% of adhesive remained on tooth; 2: More than 50% of adhesive remained on tooth; 3: All adhesive remained on tooth.

with further studies. In the present study, Transbond XT produced a higher SBS (9.86±3.2 MPa) than the other self-etch, self-adhesive resins with and without phosphoric acid etching.

Flowable composites

A plethora of flowable composite products are commercially available as bonding agents because of these favorable properties: non-stickiness and injectability[18]. On bonding orthodontic brackets to teeth, flowable composites are attractive because of their flowability and the speed at which they set and bond brackets in place on teeth[20].

Vertise Flow, powered by OptiBond adhesive technology, is a self-adhesive, light-cure flowable material which eliminates the separate steps of etching and priming prior to bonding a resin composite to dentin or enamel. Vertise Flow creates proven bonds to the tooth structure in two ways. Primarily, it is through chemical bonding between the phosphate functional groups of a glycerol phosphate dimethacrylate monomer (GPDM) and calcium ions of the tooth. Secondarily, it is through micromechanical bonding as a result of an interpenetrating network formed between the polymerized monomers of Vertise Flow and collagen fibers (as well as the smear layer) of dentin. Vertise Flow also has two methacrylate functional groups for copolymerization with other methacrylate monomers to provide increased crosslinking density and enhanced mechanical strength for the polymerized adhesive[22].

Resin cements

Self-adhesive resin cements were developed to reduce the number of application steps and technique sensitivity associated with conventional resin cements[8]. Maxcem Elite is a two-paste, dual-cure resin cement which combines the conditioning, priming, and adhesive agents into a single application. According to the manufacturer, Maxcem Elite is recommended to be used with OptiBond products for optimal bonding to enamel and dentin.

Maxcem Elite contains a proprietary redox initiator system: an efficient dual-cure mechanism that allows the resin to set quickly in the absence of light curing. Moreover, this proprietary redox initiator system eliminates the inherent discoloration of BPO/tertiary amine initiator systems for more esthetic restorations[37]. However, when compared to conventional resin cements, self-adhesive resins reportedly provided inferior bonding to both enamel[26,28,38,39] and dentin[40].

Phosphoric acid etchant versus self-etch adhesive systems

Pretreatment with 37% phosphoric acid increases bond strength[15,41] because thick outer enamel layer may prevent the permeation of self-etch primers and bonding agents, thus leaving some areas partially unetched. This then results in formation of shorter and poorly defined resin tags. By removing the outer enamel with phosphoric acid etching, longer resin tags are formed and thus bond strength is increased[42].

On the other hand, self-etch primers and bonding agents have some advantages over the phosphoric acid etchant. Self-etch primers simplify the clinical handling of adhesive systems by combining the conditioning and priming agents into a single product[2,3]. Self-etch bonding agents prevent aggressive decalcification and bulk enamel loss which are characteristics of phosphoric acid etching[43]. This means that they reduce the risk of enamel damage due to their reduced ability to sufficiently etch and penetrate the enamel surface[44]. Most self-etch adhesives did not etch enamel as deeply as the phosphoric acid etchants did, but the shallow etching pattern compromised bonding to enamel[45]. Demineralization effects of self-etch primers and bonding agents are proportional to their acidity[46]. The lower the pH, the higher the acidity, the deeper the etch; and the pH values of self-etch adhesive systems are higher than that of phosphoric acid etchant.

Self-etch adhesive systems produce high bond strengths to human coronal dentin and ground enamel surfaces[47]. With enamel, phosphoric acid pretreatment improved the bond strength; with dentin, prior acid etching was detrimental to dentin bonding effectiveness and should be avoided[26,28]. Self-etch adhesives have high viscosity, which causes an incomplete resin infiltration of the demineralized collagen network, thus resulting in inadequate dentin bonding[26]. Erhardt *et al.*[48] reported that a combination of phosphoric acid etching with acidic self-etching monomers caused sufficient high-quality hybridization of enamel and resulted in reliable bond strength on unground enamel surfaces, which could not be achieved by self-etching primers alone.

Self-adhesive flowable composites were originally designed for operative procedures that involve resin bonding. When used to bond stainless steel brackets to enamel, the early bond strength achieved by self-adhesive flowable composite was similar to that of a conventional etch-and-rinse adhesive system[49]. After thermocycling, however, the flowable composite manifested a significant decrease in its bracket retentive ability[49]. In the current study, there were differences in SBS among the self-etch, self-adhesive resin materials with and without phosphoric acid etching. SBS values of Maxcem Elite and Vertise Flow with acid etching were statistically higher than Vertise Flow without acid etching.

Bracket bond failure

Bond failure at bracket-adhesive interface, rather than at enamel-adhesive interface, is caused by the low flexural strength of composite resins[14]. Brackets bonded to enamel surfaces are temporary: they shall be removed after active treatment. Therefore, the primary orthodontic goal is to maintain sound, unblemished enamel surfaces after debonding. Enamel damage can be caused by phosphoric acid etching, bracket removal or cleaning of the teeth after debonding[45]. Phosphoric acid etching complicates the removal of residual adhesive on the enamel after debonding, subsequently leading to surface scratches, cracking, and loss of sound enamel[35,45].

ARI score "0" means that bond failure site is at adhesive-enamel interface, while score "3" means that detachment occurs at adhesive-bracket base interface. In the present study, statistically significant differences were found in the ARI scores among the test groups. This showed that two key factors heavily influenced the bonding at bracket-adhesive-enamel interfaces: type of adhesive system used and the application or omission of phosphoric acid pretreatment. ARI scores "0" and "1" were predominantly seen in Maxcem Elite and Vertise Flow groups without etching. This meant that when phosphoric acid etching was omitted, interfacial bonding between enamel and adhesive became the weakest link. However, when these adhesive materials were applied to etched enamel, their bonding strengths increased.

From the perspective of cleaning up the teeth after debonding, it is advantageous and preferred for residual adhesive to remain at bracket bases. This makes cleaning of the enamel surfaces easier and faster, with less risk of causing damage to the enamel. However, higher SBS values were found to be accompanied with higher ARI scores, which meant that a greater amount of residual adhesive remained on the teeth after debonding[50]. In the present study, Transbond XT yielded the highest SBS and higher incidences of ARI scores "2" and "3" —that is, a greater amount of residual adhesive remained on the teeth. Nonetheless, if adequate shear bond strength is attained, benefits of low ARI scores outweigh those of high ARI scores: detachment at enamel-adhesive interface lowers the risk of enamel damage and hence the probability of enamel crack formation.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. New self-etch, self-adhesive bonding systems require phosphoric acid etching prior to bonding to achieve clinically acceptable SBS values, which means that they are not successful alternatives to the traditional orthodontic bonding systems yet.
2. Further *in vivo* studies of these self-etch, self-adhesive bonding systems are needed to obtain data and findings useful and important for clinical practice, such as the effects of mineral structure of teeth and handpiece pressure on the SBS of different adhesive materials.

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