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# Cyclic shear fatigue of orthodontic brackets bonded to enamel using self-adhering flowable composites



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## **KEYWORDS**

Cvclic shear fatigue; Total-etch; Self-etch; Self-adhering flowable composites: Metal brackets

Abstract Background: Self-adhering composites are claimed to bond to teeth without prior acid etching and bonding steps. This study aimed to evaluate the cyclic shear fatigue (CSF) of metal orthodontic brackets bonded to enamel using self-adhering flowable composites (Constic and Vertise<sup>TM</sup> Flow) in comparison with that of total-etch and self-etch adhesives.

Materials and methods: Twenty-five human premolars comprising 100 surfaces were randomly divided into four groups (n = 25): total-etch, self-etch, Constic, and Vertise<sup>TM</sup> Flow. A total of 10 surfaces were used per group for baseline static shear bond strength (SSBS) evaluation and 15 surfaces for CSF evaluation. Each tooth was treated with the four bonding agents. Both SSBS and CSF were evaluated using a universal testing machine. For CSF, the staircase method was used with an initial pulling load equivalent to 60% of the SSBS of each group for 1000 cycles or until failure. The adhesive remaining index (ARI) was evaluated for surviving samples using a stereomicroscope.

Statistical analysis: A one-way ANOVA with Tukey's post hoc test was used to analyze the CSF data, and a chi-square test was used to analyze the ARI.

Results: The total-etch adhesive showed significantly higher CSF values than that of the other groups (p < 0.001). Both total-etch (10.78  $\pm$  0.31 MPa) and self-etch (6.75  $\pm$  0.91 MPa) adhesives showed significantly higher CSF than did Constic (1.94  $\pm$  0.31 MPa) and Vertise<sup>TM</sup> Flow  $(2.01 \pm 0.25 \text{ MPa}), (p < 0.001)$ . The ARI indicated that more resin remnants were observed with

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1013-9052 © 2023 The Authors. Production and hosting by Elsevier B.V. on behalf of King Saud University. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/). the total-etch and self-etch adhesives than those with the self-adhering flowable composites. However, no significant differences were observed among groups (p > 0.05).

*Conclusions:* Total-etch and self-etch adhesives showed satisfactory bond strengths for orthodontic treatment. However, Constic and Vertise<sup>TM</sup> Flow self-adhering flowable composites may not be clinically sufficient for bonding metallic orthodontic brackets to enamel.

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#### 1. Introduction

Fixed orthodontic appliances are commonly used for orthodontic treatment (Marañón-Vásquez et al., 2021). Typically, they consist of active components, including elastics and wires, and bonded components, including brackets. The force generated by elastics and wires is transmitted to the teeth through the brackets. During the treatment period, the orthodontic brackets are subjected to cyclic forces that may cause bracket failure (Daratsianos et al., 2013). Therefore, to achieve the desired treatment outcome, a durable and effective bracket–tooth bond is necessary.

Different bonding systems have been used to bond orthodontic brackets to the tooth surface, including conventional total-etch, self-etch (Bishara et al., 1998), and resinmodified glass-ionomer adhesives (Bishara et al., 2000). Total-etch adhesives can be applied in three steps (etchant, primer, and adhesive) or two steps (the primer and adhesive combined). The self-etch system can be two steps (the etchant and primer combined) or one step (all components combined), known as all-in-one (Van Meerbeek et al., 2003). There is no consensus on the ideal adhesive system for bonding orthodontic brackets to the tooth surface (Aljubouri et al., 2004; Hellak et al., 2016; Mansour & Bamashmous, 2018).

Recently, a new resin composite known as "self-adhering flowable composite" was introduced. It is a combination of the adhesive agent and resin composite and therefore eliminates the step(s) of the conventional adhesive systems. Accordingly, it minimizes technique sensitivity, reduces chair time, enhances treatment modalities, and improves patient comfort (Schauseil et al., 2016; Sabbagh et al., 2017). Examples of this composite include Vertise<sup>TM</sup> Flow (Kerr, Orange, CA, USA), Constic (DMG, Hamburg, Germany), and Fusio<sup>TM</sup> Liquid Dentin (Pentron Clinical, Orange, CA, USA) (Peterson et al., 2018). Vertise<sup>TM</sup> Flow has been formulated using the glycerophosphate dimethacrylate (GPDM) adhesive monomer. The GPDM monomer has phosphate functional groups that chemically bond to the calcium ions in tooth hydroxyapatite. It also has an acidic property required for etching tooth structures (Yuan et al., 2015). Constic contains the 10methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer, which forms a strong chemical bond with hydroxyapatite by forming 10-MDP-calcium salts (Rangappa et al., 2018). Self-adhering flowable composites are mainly used in restorations, pit and fissure sealants, liners, and blocking of undercuts (Sabbagh et al., 2017; Panse et al., 2018; Rangappa et al., 2018). The use of self-adhering composites to bond orthodontic brackets is convenient for both clinicians and patients. However, limited data are available in the literature regarding these adhesives. A few studies have evaluated their static shear bond strength (SSBS), which was found to be lower than that of conventional bonding agents, either immediately (Gungor et al., 2016) or after thermocycling (Goracci et al., 2013). However, under clinical conditions, orthodontic bond failure usually occurs because of the shear forces of the cyclic pattern. Therefore, cyclic shear fatigue (CSF) provides better information on the clinical performance of self-adhering flowable composites.

This study aimed to evaluate the CSF of orthodontic brackets bonded to the enamel of extracted human premolars using two self-adhering flowable composites in comparison to commonly used total-etch and self-etch adhesive systems. The null hypothesis was that there is no significant difference in the CSF of orthodontic brackets bonded to enamel using the conventional adhesive systems and self-adhering flowable resin composites.

#### 2. Material and methods

This study was conducted with ethical approval #189–11-89 from the ethical committee of our institute. Twenty-five human premolars comprising one hundred tooth surfaces were selected and divided into four groups according to the bonding agent used (n = 25 surfaces) as follows: total-etch adhesive, self-etch adhesive, Constic self-adhering composite, and Vertise<sup>TM</sup> Flow self-adhering composite. The composition of the materials is listed in Table 1. The brackets were bonded to each tooth on the buccal, lingual, mesial, and distal surfaces. For each group, 10 surfaces were utilized for evaluating baseline SSBS and 15 surfaces for CSF.

Teeth with signs of fracture or cracks were excluded. All teeth were scaled, polished with fine-grained pumice (Protechno, Vilamalla Girona, Spain), and washed with water. The teeth were individually embedded in acrylic resin (Hiflex RR, Prevest DenPro, Jammu, India) and stored in an incubator (Memmert GmbH, Buchenbach, Germany) at 37 °C until bonding.

#### 2.1. Bonding of orthodontic brackets

One hundred metallic brackets with slot size 0.022-inch (OC Orthodontics, McMinnville, OR, USA) were bonded to the teeth using all four tooth surfaces (Fig. 1A). Each tooth was bonded with the four different bonding systems described in section 2. The bonding technique of each adhesive system was applied according to the manufacturers' recommendations and in a rotational pattern in the following order: buccal, mesial, lingual, and distal surfaces. For instance, if the total-etch adhesive was used on the buccal surface of one tooth, it would be used on the mesial surface of the next one, and so on. Premolar brackets were used for buccal and lingual surfaces, whereas lower incisor brackets were used for mesial

**Table 1** Materials used in the study according to the manufacturers and the study by Rangappa et al., 2018.

Bonding system	Composition	Manufacturer
Single Bond universal adhesive	10-MDP phosphate monomer, Vitrebond copolymer, HEMA, dimethacrylate resins, filler, silane, initiators, ethanol, water	3 M Unitek, Monrovia, CA, USA
Transbond XT light cure paste	Silane treated quartz, Bisphenol A Diglycidyl Ether Dimethacrylate (BISGMA), Bisphenol A Dimethacrylate, silane treated silica, Diphenyliodonium Hexafluorophosphate, Triphenylantimony	3 M Unitek, Monrovia, CA, USA
Vertise Flow	Glycero-phosphate dimethacrylate (GPDM), Prepolymerized filler containing barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride.	Kerr, Orange, CA, USA
Constic	Methacrloxydecyl dihydrogen phosphate (MDP), BISGMA, ethoxylated bisphenol A dimethacrylate (EBADMA), urethane dimethacrylate, 2- hydroxy ethyl methacrylate (HEMA), triethylene glycol dimethacrylate (TEGDMA), and 1,6-hexanediol dimethacrylate (HDMA) monomers.	DMG, Hamburg, Germany

and distal surfaces. In each testing cycle, a group of samples was bonded and stored in silane for 24 h until testing. The bonding was performed by a single operator.

## 2.1.1. Bonding techniques

The application steps for the different bonding systems are described in Table 2.

## 2.2. Static shear bond strength

The SSBS was evaluated using a universal testing machine (Multitest 2.5-i, Mecmesin, UK) (Fig. 1B-D). The teeth were fixed at the lower stage, and a ligature wire was tied between the brackets and a hook attached to a 500 N load cell. The tests were performed at a pull speed of 2 mm/min. The mean SSBS of each bonding system was calculated by dividing the force by the apparent surface area of the brackets (9 mm<sup>2</sup> and 7.5 mm<sup>2</sup> for premolar and lower incisor brackets, respectively). The mean force at failure was used to calculate the starting load for the CSF test (60% of the mean baseline SSBS value).

# 2.3. Cyclic shear fatigue

The CSF was tested using the same setup described in section 2.2 for the SSBS. The test was performed with an initial pulling load for each group that was equal to 60% of the corresponding static force at failure. The speed was set at 2 Hz, and the vertical traveling distance was 2 mm for 1000 cycles or until the bond failed. A staircase method was followed in which if a bracket survived 1000 cycles, the load was increased by 20% for the next sample; however, if a bracket did not survive 1000 cycles, the load was reduced by 20% for the next sample. For each subsequent sample, the process of elevating and



**Fig. 1** Sample testing set up. (A) Orthodontic brackets bonded to buccal, lingual, mesial, and distal surfaces of the teeth. (B) Shear bond strength testing machine. (C) Sample attached to the machine. (D) Sample after de-bonding of orthodontic brackets.

**Table 2**Application steps for the bonding systems.

Bonding system	Application steps	
Total-Etch	<ol> <li>Etch enamel with 37% phosphoric acid (3 M Unitek, Monrovia, CA, USA) for 30 s then rinse with water spray for 5 s and air-dry for 5 s.</li> <li>Apply Single Bond Universal adhesive to</li> </ol>	
	the tooth surface using a disposable appli- cator, rub for 20 s, and then apply a gentle stream of air for 5 s.	
	3- Light cure for 10 s using LED light cure (Elipar DeepCure-S, 3 M Unitek, Mon- rovia, CA, USA).	
	4- Apply Transbond XT light cured paste to the metal bracket then place it on the tooth with steady slow pressure and remove excess material.	
	5- Light cure the cement for 15 s from the mesial and distal sides.	
Self-Etch	The same bonding technique as described for self- etch adhesives but without prior acid etching	
ConsticVertise Flow	<ol> <li>Dry the enamel gently with air.</li> <li>Apply Constic or Vertise<sup>TM</sup> Flow self- adhering composite on the bracket using Luer-Lock-Tip and then press it on the tooth surface with gentle pressure for 25 s.</li> </ol>	
	3- Remove excess composite and then light cure for 20 s.	

reducing the load by 20% following each sample was repeated until all samples in each group were tested (Mansour et al., 2011).

#### 2.4. Survival % and adhesive remnant index

The assessment of survival % (i.e., samples that survived the cyclic fatigue testing) was based on the occurrence event; bond failure was scored as 1, while no bond failure was scored as 0. For all failed samples after CSF, enamel surfaces were examined under a digital stereomicroscope (RaySmart Technology Co., Ltd., Shenzhen, China) at 30x magnification to assess the adhesive remnant index (ARI). The samples were scored from 0 to 3 according to the following criteria: 0, 0% adhesive remaining on the enamel; 1, < 50% adhesive remaining on the enamel; and 3, 100% adhesive remaining on the enamel (Hellak et al., 2016).

#### 2.5. Scanning electron microscopy

One failed sample from each group was tested under a scanning electron microscope (SEM) (Aura100, Seron Technologies, Gyeonggi-do, Korea) at 100x magnification.

#### 2.6. Statistical analyses

Descriptive analyses of the collected data were performed. One-way ANOVA was used to determine the effects of the different adhesive systems on the CSF. Linear regression was used to determine the significance of the independent variables (different adhesive systems and/or bracket sites) on the dependent variable (CSF). Descriptive statistics and the chi-square test were used for the ARI data. All statistical tests were performed using  $\alpha$  at p < 0.05.

#### 3. Results

#### 3.1. Cyclic shear fatigue

The CSF results along with the baseline SSBS are presented in Table 3. The highest CSF score was recorded in the total-etch group, which was statistically different from that of the other groups (p < 0.001). The self-etch adhesive also had a significantly higher bond strength than did the self-adhering composite (p < 0.001). There was no significant difference in bond strength between the Vertise<sup>TM</sup> Flow and Constic self-adhering flowable composites (p = 0.97). Therefore, the null hypothesis was rejected.

Although the main dependent variable in this experiment was the type of adhesive system, the bracket site was considered another dependent variable. Therefore, a linear regression analysis was performed to determine the effect of the bracket site and/or type of adhesive system on the SBS and CSF. Linear regression analysis data with  $R^2 = 0.799$  showed that the type of adhesive had a statistically significant effect on the CSF (p < 0.001), whereas the bracket site had no effect (p = 0.588).

## 3.2. Survival % and adhesive remnant index

The survival data and ARI scores of the samples that failed fatigue testing are listed in Table 4. The total-etch group showed the highest survival rate (66.7%), whereas the Vertise<sup>TM</sup> Flow self-adhering composite group had the lowest survival rate (46.7%). Both the self-etch and Constic self-adhering composite groups showed the same survival rate (53.3%).

The ARI score for the total-etch group was predominantly 1, with one sample scoring 2. Similarly, the score for the selfetch group was predominantly 1, with two samples scoring 0 and 2. The ARI scores for the Constic and Vertise<sup>TM</sup> Flow self-adhering composites were evenly divided between 0 and 1. However, there was no statistically significant difference among the different types of adhesive systems (p < 0.55). Stereomicroscopic and SEM images of selected samples with different ARI scores are shown in Fig. 2.

 Table 3
 Cyclic shear fatigue of the adhesive systems and baseline SSBS. Superscript letters represent post hoc Tukey test significant pairs.

Bonding System	CSF (MPa)	Baseline SSBS (MPa)
a. Total-Etch	$10.78 \pm 3.66^{\mathrm{b,c,d}}$	9.87
b. Self-Etch	$6.75~\pm~0.91~^{ m a,c,d}$	9.13
c. Constic	$1.94 \pm 0.31^{a,b}$	4.59
d. Vertise <sup>TM</sup> Flow	$2.01~\pm~0.25~^{\rm a,b}$	3.76

Adhesive System N	Ν	Survived (%)	ARI Scores of failed samples			
		0 (%)	1 (%)	2 (%)	3 (%)	
Total-Etch	15	10 (66.7)	0 (0.0)	4 (80.0)	1 (20.0)	0 (0.0)
Self-Etch	15	8 (53.3)	1 (14.3)	5 (71.4)	1 (14.3)	0 (0.0)
Constic	15	8 (53.3)	3 (42.9)	4 (57.1)	0 (0.0)	0 (0.0)
Vertise <sup>TM</sup> Flow	15	7 (46.7)	4 (50.0)	4 (50.0)	0 (0.0)	0 (0.0)

#### 4. Discussion

In the present study, human premolars were used. Bonding was performed on all tooth surfaces in a consequential manner. Due to different available bonding areas, incisor brackets were used for proximal surfaces, and premolar brackets were used for buccal and lingual surfaces. To validate this methodology, linear regression was performed and showed that the bracket site has no significant effect on the CSF.

Under clinical conditions, orthodontic tooth bond failure usually occurs at a force lower than the SSBS. Therefore, the CFS tests provide more reliable data than does SSBS (Soderquist et al., 2006; Abdelnaby, 2011). In our study, the CFS was evaluated using the staircase method. This technique provides a reasonable measure of the mean fatigue limit of each group and allows evaluation of the standard deviation around the mean stress while it requires a smaller sample than other methods (Daratsianos et al., 2013; Mansour & Bamashmous, 2018).



Fig. 2 Stereomicroscopic (30x) and SEM (100x) images of enamel surface after debonding. (A and B) ARI score 0: no adhesive remained on the tooth. (C and D) ARI score 1: < 50% of adhesive remained on the tooth. (E and F) ARI score 2: > 50% of adhesive remained on the tooth. The impression of the bracket base was evident on the enamel surface of samples scored 1 and 2. Images b, d, and f are the corresponding SEM images of the samples B, D, and F, respectively.

As reported in the literature, although controversial, the minimum shear bond strength required for orthodontic brackets to withstand the occlusal forces ranges between 6 and 8 MPa (Reynolds, 1975). According to the results of the present study, the CSF of both total-etch (10.78 MPa) and selfetch (6.75 MPa) composites were in the acceptable range for orthodontic treatment, whereas the self-adhering flowable composites had CSF below the minimum required strength (1.94 MPa and 2.01 MPa for Constic and Vertise<sup>TM</sup> Flow, respectively).

The CSF results revealed that the total-etch group had a significantly higher bond strength than the self-etch group, which is consistent with the results of the study by Mansour et al. (2011). Self-etch adhesives rely on MDP monomers, which are responsible for etching the enamel surface (Carrilho et al., 2019). The higher strength of the total-etch composite may be attributed to the better ability of phosphoric acid in total-etch adhesives to etch hypermineralized enamel compared to the lower pH monomer of the self-etch adhesive, leading to a significantly higher bond strength (Cerone et al., 2019). In contrast, several in vitro studies (Arnold et al., 2002; Hellak et al., 2016) and clinical trials (Aljubouri et al., 2004; Reis et al., 2008; Farhadian et al., 2019) have shown comparable bond strengths between the total-etch and self-etch adhesives.

Similarly, the lower bond strength of the self-adhering composites may indicate inadequate etching of the enamel surface. Owing to the recent introduction of self-adhering flowable composites, only a few studies have evaluated these materials for bonding orthodontic brackets. Similar to the present study's results, Gungor et al. (2016) found that the bond strength of Vertise<sup>TM</sup> Flow was significantly lower than that of the total-etch and self-etch adhesives. Goracci et al. (2013) reported that after thermocycling, Vertise<sup>TM</sup> Flow had a significantly lower SSBS than those of the total-etch and selfetch adhesives. The authors, therefore, questioned the durability of the self-adhering flowable composites. As restorative materials, it was demonstrated that the self-adhering flowable composites had a significantly lower bond strength to enamel than that of conventional flowable composites bonded with total-etch techniques (Peterson et al., 2018). It may be inferred that the combination of all components into the self-adhering flowable composite interferes with the action of the functional monomers.

After cyclic fatigue, the brackets are expected to fail at lower stress because of the presence of defects, such as air voids, cracks, inhomogeneity in resin thickness, rough bracket bases, and enamel surfaces, which will affect the stress distribution pattern, resulting in failure (Yan et al., 2022). Accordingly, we inferred that the inhomogeneity at the adhesive interface can be reduced using the total-etch adhesive. Additionally, impurities in functional monomers can affect the durability of the enamel bond (Yoshihara et al., 2015), which may explain the CSF values of the self-etch adhesive and self-adhering flowable composites.

The ARI provides useful clinical information about the quality of the bond. Besides having an adequate bond strength, the ideal adhesive is desirable to fail at the cement-enamel interface which allows easy removal after debonding without causing enamel fracture (Reynolds, 1975).

In the current study, the ARI showed an association between the amount of remaining composite and the bonding system used. However, there was no significant difference among groups, which may be attributed to the limited number of samples that failed after cyclic fatigue. The ARI scores were predominantly 1 and 2 in the total-etch and self-etch adhesive groups indicating that the failure occurred mainly at the adhesive-bracket interface. These findings are congruent with those of Mansour et al. (2011); Farhadian et al. (2016); Hellak et. Al. (2016). The self-adhering flowable composites had ARI scores 0 and 1, where 50% of the samples showed adhesive failure at the resin-enamel interface due to insufficient bond strength, which corroborates the results of Gungor et al. (2016). The fact that less residual resin after debonding is an advantage for orthodontic adhesives, self-adhering composites could be used in orthodontics if their bond strength is improved.

#### 5. Conclusion

The total-etch and self-etch adhesives showed clinically sufficient CSF of orthodontic brackets to enamel, whereas the CSF of self-adhering flowable composites was significantly lower and clinically insufficient. The ARI scores showed that the total-etch and self-etch adhesives failed mainly at the adhesive-bracket interface, whereas the self-adhering flowable compsites fail frequently at the resin-enamel interface.

#### 6. Authorship declaration

All authors have contributed significantly and agree with the present manuscript.

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#### Ethical statement

The study was conducted after getting an ethical approval #189–11-19 from the Research and Ethics Committee at the Faculty of Dentistry, King Abdul Aziz University.

## CRediT authorship contribution statement

Tariq Abu Haimed: Conceptualization, Methodology, Validation, Resources, Writing – original draft, Writing – review & editing, Supervision, Project administration. Reham Osama Filfilan: Methodology, Investigation, Resources, Writing – original draft. Mawadah Nazih Hassoubah: Methodology, Investigation, Resources, Writing – original draft. Ensanya A. Abou Neel: Writing – original draft, Writing – review & editing. Mohamed Bamashmous: Conceptualization, Methodology, Formal analysis, Supervision.

#### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.sdentj.2023.07.014.

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