

Comparison of shear bond strengths of orthodontic brackets bonded with flowable composites

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This study evaluated the shear bond strengths of orthodontic brackets bonded to human premolars using five different combinations of flowable composites and one-step self-etching adhesives ($n=12$): (1) Adper Easy Bond+Filtek Supreme XT Flow; (2) Futurabond NR+Grandio Flow; (3) Clearfil S3 Bond+Clearfil Majesty Flow; (4) AdheSE One+Tetric EvoFlow; and (5) Transbond Plus Self Etching Primer+Transbond XT Light Cure Adhesive. After shear bond strength testing, adhesive remnant index (ARI) scores were given according to the amount of adhesive and resin remaining on the brackets. On shear bond strength, there were no statistically significant differences between Groups 2 and 4 and between Groups 3 and 5 ($p>0.05$). On ARI scores, the predominant ARI scores in Groups 1, 2, 3, and 5 were 4, 2, 5, and 4 respectively; in Group 4, they were 0 and 4. Results showed that some combinations of flowable composites and self-etching adhesives might not be suitable for orthodontic use due to their low shear bond strengths and high ARI scores—with the latter signaling the risk of damaging the enamel surface during debonding.

Keywords: Orthodontic bracket, Flowable composite, Self-etching adhesive, Shear bond strength

INTRODUCTION

Acid-etch bonding is now an accepted and widely used technique in adhesive and preventive dentistry, characterized by the formation of tag-like resin extensions into the enamel micropores¹. Among the many and varied application areas this technique has found itself, one of which is the bonding of orthodontic brackets to enamel²⁻⁴.

On orthodontic attachments, metal brackets have been the traditional choice of orthodontists. However, with a heightened awareness on esthetics in recent years, some patients have opted for ceramic brackets instead of metal ones. Each type of orthodontic brackets has its own pros and cons. With ceramic brackets, a key problem is that they do not bend during debonding, thereby causing and creating cracks in the enamel surface. With metal brackets, a key problem is inadequate bond strength to enamel such that they are not able to withstand forces applied during orthodontic treatment⁵⁻⁷. Although varied bond strength values of brackets to enamel have been suggested by different studies, the ideal bond strength should be less than the breaking strength of enamel which is approximately 14 MPa⁸.

In the bonding of orthodontic brackets to the enamel surface, a conventional adhesive system comprises three separate agents: an enamel conditioner, a primer solution, and an adhesive resin⁹. To reduce chairtime, simplified adhesives—often referred to as self-etching adhesives—were developed subsequently. An apparent advantage of the self-etch adhesive systems is their simple application technique, since they combine—in

one solution—the etching, priming, and bonding steps of the traditional etch-and-rinse adhesives¹⁰. Other advantages offered by the self-etching systems include reduced technique sensitivity (because they eliminate the dependence on moist bonding which is characteristic of etch-and-rinse adhesive systems^{11,12}) and providing acceptable shear bond strength (SBS) values for bracket bonding¹³⁻¹⁵.

Apart from the influences of orthodontic brackets and adhesive systems in an orthodontic bonding procedure, composite resins also play an important role in bonding results. Recently, flowable resins were introduced to the dental market with claims of greater fluidity and elasticity, better adaptation to internal cavity walls, and easier insertion than previously available products. These materials either have a lower filler loading or a greater proportion of diluent monomers in their formulations¹⁶. As to their use and applicability in orthodontics, a survey of the published literature revealed little information. The few studies which were conducted had flowable composites used in combination either with a commonly used orthodontic adhesive Transbond XT or without an adhesive^{17,18}.

To the best of our knowledge, no studies have been undertaken to examine the bond strength of flowable composites used in combination with one-step self-etching adhesives. Therefore, the aim of this study was to investigate the bond strengths of flowable composites used in combination with their respective one-step self-etching adhesives and then compare these SBS values against that obtained with a conventional orthodontic self-etching primer and adhesive paste. In addition, a modified adhesive remnant index (ARI) was

used to determine the amount of adhesive and resin remained on the bracket surfaces.

MATERIALS AND METHODS

Preparation of tooth specimens

Sixty human premolars extracted for orthodontic purposes were collected and stored in deionized water. Teeth with hypoplastic enamel, fractures or caries were excluded from the study. Each tooth was mounted vertically in a self-cure acrylic resin so that the crown was exposed. The teeth were cleaned and polished with a slurry of nonfluoridated flour of pumice (Moyco Industries, Philadelphia, PA) for 10 seconds by using a rubber prophylactic cup, and then rinsed with a stream of water for 10 seconds.

Orthodontic bonding

Stainless steel premolar brackets (Generous Roth Brackets, GAC International Inc., Islandia, NY), with an average bracket base surface area of 12.13 mm², were used for all teeth. With 12 premolars in each group, the teeth were randomly divided into five test groups as follows:

- Group 1: Adper Easy Bond+Filtek Supreme XT Flow (3M ESPE, St Paul, USA);
- Group 2: Futurabond NR+Grandio Flow (VOCO, Cuxhaven, Germany);
- Group 3: Clearfil S3 Bond+Clearfil Majesty Flow (Kuraray Medical Inc., Tokyo, Japan);
- Group 4: AdheSE One+Tetric EvoFlow (Ivoclar Vivadent, Schaan, Liechtenstein);
- Group 5: Transbond Plus Self Etching Primer+Transbond XT Light Cure Adhesive Paste (3M Unitek, Monrovia, CA, USA).

The compositions of the adhesive systems and resin composites used are given in Tables 1 and 2. In each test group, the brackets were bonded according to each manufacturer's instructions. Light curing was performed with a halogen curing light (Optilux 501, Kerr Corp., Orange, CA, USA) from the mesial and distal sides of the brackets, at a duration of 20 seconds from each side. After light curing, all samples were stored in 37°C deionized water for 24 hours.

Shear bond strength test

For SBS testing, a mounting jig was used to align the facial surface of each tooth to be perpendicular to the bottom of the mold. Each tooth was oriented with the testing device as a guide such that its facial surface was parallel to the applied force during shear bond strength testing.

A steel rod with one flattened end was attached to the crosshead of a Zwick test machine (Zwick Test Machine, Zwick GmbH & Co., Ulm, Germany). An occlusogingival load was applied to the bracket, producing a shear force at the bracket-tooth interface. SBS values were measured at a crosshead speed of 5 mm/min, and a computer connected to the Zwick test machine recorded the result of each test. The force required to shear off the bracket was directly recorded in newtons (N) and converted into megapascals (MPa) using this equation, Shear force (MPa)=Debonding force (N)/Bracket surface area (mm²), where 1 MPa=1 N/mm².

Adhesive remnant index (ARI) scoring

After debonding, the teeth and brackets were examined under a stereomicroscope (Leica MS5, Singapore) at 16× magnification. Modified ARI scores¹⁹⁾ were given based on the amount of adhesive and flowable resin remaining

Table 1 Compositions of the composite resins used in this study

Product	Composition*	Filler weight (%)	Manufacturer
Filtek Supreme XT Flow	Bis-GMA, TEGDMA, Bis-EMA silica nanofiller, zirconia nanofiller	65%	3M ESPE, St. Paul, USA
Grandio Flow	Bis-GMA, TEGDMA, HDDMA, SiO ₂ nanofillers, initiators, stabilizers	80%	VOCO, Cuxhaven, Germany
Clearfil Majesty Flow	TEGDMA, silanated barium glass filler, silanated colloidal silica, hydrophobic aromatic dimethacrylate, dl-camphorquinone	48%	Kuraray Medical Inc., Tokyo, Japan
Tetric EvoFlow	Bis-GMA, urethane dimethacrylate, decandiol dimethacrylate, barium glass filler, ytterbium trifluoride, mixed oxide, silicon dioxide, prepolymer additives, catalysts, stabilizers, pigments	81%	Ivoclar Vivadent, Schaan, Liechtenstein
Transbond XT Light Cure Adhesive Paste	Silane-treated quartz, bisphenol A diglycidyl ether dimethacrylate, bisphenol A bis(2-hydroxyethyl ether) dimethacrylate, silane-treated silica	77%	3M Unitek, Monrovia, CA, USA

*Bis-GMA: Bisphenol-A-diglycidyl methacrylate; Bis-EMA: Ethoxylated bisphenol-A-glycol dimethacrylate; HDDMA: 1,6-Hexanediol dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate.

Table 2 Compositions of the one-step self-etching adhesives used in this study

Product	Composition*	Manufacturer
Adper Easy Bond	HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid, silica filler, ethanol, water, initiators, stabilizers	3M ESPE, St. Paul, USA
Futurabond NR	Bis-GMA, HEMA, phosphate methacrylates, BHT, ethanol, fluorides, CQ, silicium dioxide nanoparticles	VOCO, Cuxhaven, Germany
Clearfil S3 Bond	MDP, Bis-GMA, HEMA, photoinitiators, ethanol, water, silanated colloidal silica	Kuraray Medical Inc., Tokyo, Japan
AdheSE One	Bis-Acrylamide, water, bis-methacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxyl alkyl methacrylamide, highly dispersed silicon dioxide, catalysts, stabilizers	Ivoclar Vivadent, Schaan, Liechtenstein
Transbond Plus Self Etching Primer	Methacrylate ester derivative, water	3M Unitek, Monrovia, CA, USA

*Bis-GMA: Bisphenol-A-diglycidyl methacrylate; MDP: 10-Methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-Hydroxyethyl methacrylate; BHT: Butylhydroxytoluene; CQ: Camphorquinone.

on the bracket surface. ARI scores ranged from 5 to 0, where 5=100% of adhesive and flowable resin remained on the bracket, 4=100%–75% of adhesive and flowable resin remained on the bracket, 3=75%–50% of adhesive and flowable resin remained on the bracket, 2=50%–25% of adhesive and flowable resin remained on the bracket, 1=less than 25% of adhesive and flowable resin remained on the bracket, 0=No adhesive and flowable resin remained on the bracket.

Statistical analysis

For shear bond strength, descriptive statistics — including the mean, standard deviation, and minimum and maximum values— were calculated for each test 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. For ARI scores, they were evaluated using Fisher's exact test in $m \times n$ cross tables. Statistical significance was determined at a probability value of $p < 0.05$.

RESULTS

The SBS values of all test groups are presented in Fig. 1. Group 5 had the highest bond strength but did not differ significantly from Group 3. There were no statistically significant significances between Groups 2 and 4 as well as between Groups 1 and 3 ($p > 0.05$).

The frequency distribution of ARI scores for all the test groups is shown in Fig. 2. Fisher's exact test showed statistically significant differences among all groups ($p < 0.05$). In Groups 1, 2, 3, and 5, the predominant

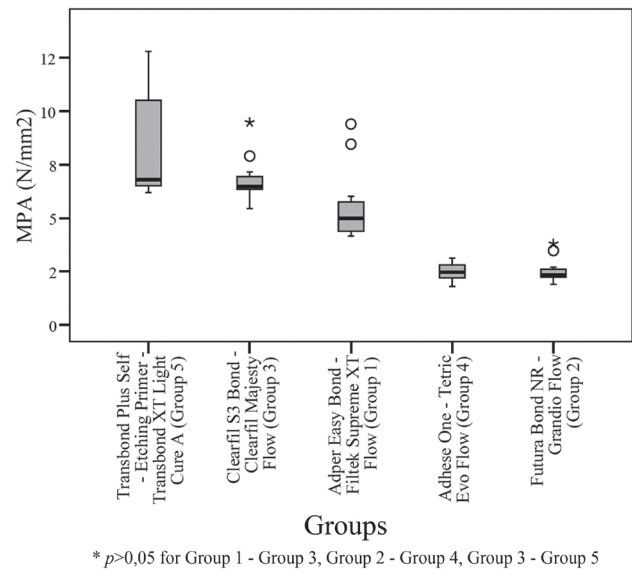


Fig. 1 Schematic diagram of the mean SBS values (MPa) of all test groups and their standard deviations.

failure scores were 4, 2, 5, and 4, respectively. In Group 4, the predominant failure scores were 0 and 4.

DISCUSSION

To date, the conventional bracket bonding system is Transbond XT, which comprises an acid gel, a primer, and an adhesive paste. Being the traditional choice of orthodontists to bond brackets to the enamel surface,

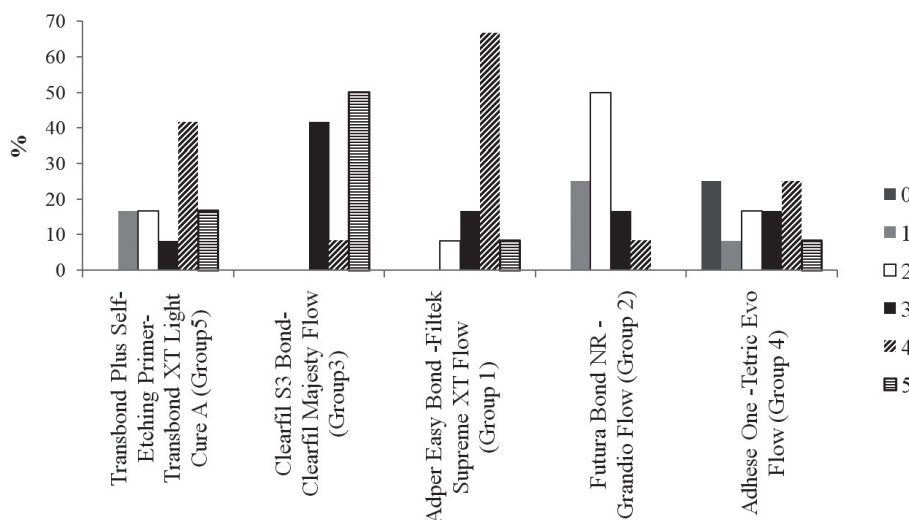


Fig. 2 Schematic diagram of the frequency distribution of Adhesive Remnant Index (ARI) scores among the test groups.

Transbond XT was also the gold standard used in many SBS studies which assessed the orthodontic bonding effectiveness of these new products^{20,21}. In addition to Transbond XT, Transbond Plus Self Etching Primer was recently introduced and found to produce a bond strength close to that of Transbond XT²². Therefore, in this study, a combined use of Transbond Plus Self Etching Primer and Transbond XT Light Cure Adhesive Paste served as the ‘standard’ against which the SBS and ARI results of all the other test groups were compared.

On the minimum bond strength required for bonding brackets, it was suggested to range between 6 and 8 MPa²³. On the maximum bond strength, it should be less than the breaking strength of enamel, which is about 14 MPa⁸. Based on this data range, we thus concluded that Transbond Plus Self Etching Primer and Clearfil S3 Bond were suitable for orthodontic bonding use.

Transbond Plus Self Etching Primer had the lowest pH value among all the adhesive systems tested in this study: Adper Easy Bond pH=2.4, Futurabond NR pH=1.4, Clearfil S3 Bond pH=2.7, AdheSE One pH=1.5, Transbond Plus Self Etching Primer pH=1. However, the high bond strength of Transbond Plus Self Etching Primer did not stem from its high acidity, as it was recently demonstrated that the pH value of self-etching adhesives is not a primary determinant of SBS²⁴. As for Transbond XT, the high bond strength it produced was probably due to the higher consistency of its adhesive paste as compared to other flowable composites. Although flowable composites easily spread and adapt to the bracket base, downward flow of material due to gravity is a critical disadvantage¹⁷. It was likely that the downward flow of flowable composites resulted in poor adaptation of the composites with the bracket base, whereas the higher consistency of Transbond XT Adhesive Paste enabled the bracket to be placed onto the

tooth surface in its desired position.

Compared to phosphoric acid, self-etching adhesives boast of several advantages. First and foremost, self-etching adhesives prevent excessive decalcification—which is characteristic of phosphoric acid etching¹⁵. In the same vein, self-etching adhesives reduce the risk of enamel damage due their reduced ability to penetrate the enamel surface^{9,13}. Published studies thus far have used self-etching adhesives in combination with traditional orthodontic pastes and reported that adequate bond strengths were produced for bonding brackets^{15,25}. However, today’s dental market sees a constant stream of newly developed bonding agents and composite resins, leading to a constant debate on whether these new products could be leveraged and adopted for orthodontic uses. Against this background, the present study took a different approach of using self-etch adhesives with their respective flowable resins instead of orthodontic pastes, in a bid to explore the potential and feasibility of using these newly developed products for orthodontic bonding.

To the best of our knowledge, no studies have been undertaken to investigate the orthodontic bonding effectiveness of one-step self-etching adhesives with their respective flowable resins. This meant that the SBS results of the present study could not be compared with any published reports. As to the explanation of the SBS results achieved in this study, no specific correlations could be made between SBS and quantifiable factors such as resin type or filler load. It was previously stated by Park *et al.*¹⁷ that in addition to the lack of clear information on the exact volume of monomer in each product, the monomers could be in the form of a co-mixture of monomers, thereby making it difficult to establish any correlation with the SBS results. As to the low bond strengths obtained in Groups 1, 2, and 4, it could be attributed to the complicated interactions at the

tooth-adhesive-bracket interfaces which could adversely affect bond strength¹⁷.

Further on the SBS results obtained in this study, it should be mentioned that they were obtained by means of *in vitro* experiments carried out in the laboratory. However, laboratory conditions might not faithfully mimic the clinical environment. For instance, stringent tooth selection criteria were used in this study whereby teeth with enamel defects or fractures were eliminated. In actual clinical situations, not-so-perfect teeth are the ones typically encountered. Besides, patient-based factors such as tooth-brushing or oral habits could affect treatment outcome. Therefore, long-term clinical studies are required to verify the bonding effectiveness of the products tested in this study under laboratory conditions.

Another factor that affects the bond strength of resin-based materials to dental hard tissues is the effect of composite aging. The most commonly used artificial aging methods in the laboratory are long-term water storage and thermocycling. The ISO TR 11450 standard (1994) recommends 500 cycles in water between 5 and 55°C. However, doubts were raised on the sufficiency of this number of thermocycles to affect bond strength^{26,27}. On one hand, the shear bond strengths of self-etching adhesives were found to decrease with an increase in the number of thermocycles and/or storage period^{28,29}. On the other hand, a recent study showed that 2 years of storage and 6,000 times of thermocycling had no significant effect on the shear bond strength of self-etching adhesives³⁰. In light of these contradicting reports, further *in vitro* research is necessary to evaluate the bonding durability of flowable composites before clinical use.

After SBS testing, it is expedient to determine the site of material failure and give the appropriate ARI scores²⁰. Material failure at the bracket-adhesive interface, rather than the enamel-adhesive interface, is caused by the low flexural strength of composite resin²¹. In the present study, however, ARI scores of 4 and 5 were predominant for the flowable composites Filtek Supreme XT Flow and Clearfil Majesty Flow. Both Filtek Supreme XT Flow and Clearfil Majesty Flow had lower filler loadings of 65% and 48% respectively, hence accounting for their low flexural strengths. On the contrary, as for flowable composites with a higher filler load, namely Grandio Flow and Tetric EvoFlow, they had more ARI scores of 0-2 when compared to the other test groups. Based on these ARI scores, it became apparent that the clinical use of flowable composites with high ARI scores for orthodontic bracket bonding remained a concern as they pose a risk of damaging the enamel surface during debonding¹⁸.

CONCLUSION

Notwithstanding the limitations of this study, it seemed reasonable to conclude that some flowable composites and their respective one-step self-etching adhesives might not be suitable for bonding of orthodontic brackets. Therefore, clinicians must be discerning and cautious

that materials selected for orthodontic bonding must yield adequate SBS and a low ARI score to avoid damaging the enamel surface.

ACKNOWLEDGMENTS

The authors are grateful to Dr. Erdem Karabulut for his help and support with the statistical analysis.

REFERENCES

- 1) Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1955; 34: 849-853.
- 2) Bishara SE, Laffoon JF, Vonwald L, Warren JJ. The effect of repeated bonding on the shear bond strength of different orthodontic adhesives. *Am J Orthod Dentofacial Orthop* 2002; 121: 521-525.
- 3) Newman GV, Snyder WH, Wilson CE Jr. Acrylic adhesives for bonding attachments to tooth surfaces. *Angle Orthod* 1968; 38: 12-18.
- 4) Retief DH, Dreyer CJ, Gavron G. The direct bonding of orthodontic attachments to teeth by means of an epoxy resin adhesive. *Am J Orthod* 1970; 58: 21-40.
- 5) Uysal T, Ustidal A, Nur M, Catalbas B. Bond strength of ceramic brackets bonded to enamel with amorphous calcium phosphate-containing orthodontic composite. *Eur J Orthod* 2010; 32: 281-284.
- 6) Bishara SE. Ceramic brackets and the need to develop national standards. *Am J Orthod Dentofacial Orthop* 2000; 117: 595-597.
- 7) Verstryngge A, Ghesquiere A, Willems G. Clinical comparison of an adhesive precoated vs. an uncoated ceramic bracket system. *Orthod Craniofac Res* 2004; 7: 15-20.
- 8) Retief DH. Failure at the dental adhesive-etched enamel interface. *J Oral Rehabil* 1974; 1: 265-284.
- 9) Bishara SE, Oonsombat C, Ajlouni R, Laffoon JF. Comparison of the shear bond strength of two self-etch primer/adhesive systems. *Am J Orthod Dentofacial Orthop* 2004; 125: 348-350.
- 10) Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. *Dent Mater* 2005; 21: 864-881.
- 11) Perdigo J, Anauate-Netto C, Carmo AR, Lewgoy HR, Cordeiro HJ, Dutra-Corrêa M, Castilhos N, Amore R. Influence of acid-etching and enamel beveling on the 6-month clinical performance of a self-etch dentin adhesive. *Compend Contin Educ Dent* 2004; 25: 33-40.
- 12) Christensen GJ. Preventing postoperative tooth sensitivity in Class I, II and V restorations. *J Am Dent Assoc* 2002; 133: 229-231.
- 13) Eminkahyagil N, Korkmaz Y, Gokalp S, Baseren M. Shear bond strength of orthodontic brackets with newly developed antibacterial self-etch adhesive. *Angle Orthod* 2005; 75: 843-848.
- 14) Cal-Neto JP, Carvalho F, Almeida RC, Miguel JA. Evaluation of a new self-etching primer on bracket bond strength *in vitro*. *Angle Orthod* 2006; 76: 466-469.
- 15) Arhun N, Arman A, Sesen C, Karabulut E, Korkmaz Y, Gokalp S. Shear bond strength of orthodontic brackets with 3 self-etch adhesives. *Am J Orthod Dentofacial Orthop* 2006; 129: 547-550.
- 16) Payne JH IV. The marginal seal of Class II restorations: flowable composite resin compared to injectable glass ionomer. *J Clin Pediatr Dent* 1999; 23: 123-130.
- 17) Park SB, Son WS, Ko CC, García-Godoy F, Park MG, Kim HI, Kwon YH. Influence of flowable resins on the shear bond

- strength of orthodontic brackets. Dent Mater J 2009; 28: 730-734.
- 18) Ryou DB, Park HS, Kim KH, Kwon TY. Use of flowable composites for orthodontic bracket bonding. Angle Orthod 2008; 78: 1105-1109.
 - 19) Romano FL, Correr AB, Sobrinho LC, Magnani MBBA, Siqueira VCV. Shear bond strength of metallic brackets bonded with a new orthodontic composite. Braz J Oral Sci 2009; 8: 76-80.
 - 20) Shammaa I, Ngan P, Kim H, Kao E, Gladwin M, Gunel E, Brown C. Comparison of bracket debonding force between two conventional resin adhesives and a resin-reinforced glass ionomer cement: an *in vitro* and *in vivo* study. Angle Orthod 1999; 69: 463-469.
 - 21) Minick GT, Oesterle LJ, Newman SM, Shellhart WC. Bracket bond strengths of new adhesive systems. Am J Orthod Dentofacial Orthop 2009; 135: 771-776.
 - 22) Romano FL, Tavares SW, Nouer DF, Consani S, Borges de Araújo Magnani MB. Shear bond strength of metallic orthodontic brackets bonded to enamel prepared with self-etching primer. Angle Orthod 2005; 75: 849-853.
 - 23) Reynolds IR. A review of direct orthodontic bonding. Br J Orthod 1975; 2: 171-178.
 - 24) Ostby AW, Bishara SE, Denehy GE, Laffoon JF, Warren JJ. Effect of self-etchant pH on the shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 2008; 134: 203-208.
 - 25) Attar N, Taner TU, Tülümen E, Korkmaz Y. Shear bond strength of orthodontic brackets bonded using conventional vs one and two step self-etching/adhesive systems. Angle Orthod 2007; 77: 518-523.
 - 26) Hasegawa T, Retief DH, Russell CM, Denys FR. Shear bond strength and quantitative microleakage of a multipurpose dental adhesive system resin bonded to dentin. J Prosthet Dent 1995; 73: 432-438.
 - 27) Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent 1999; 27: 89-99.
 - 28) Cehreli ZC, Kecik D, Kocadereli I. Effect of self-etching primer and adhesive formulations on the shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 2005; 127: 573-579.
 - 29) Oesterle LJ, Shellhart WC. Effect of aging on the shear bond strength of orthodontic brackets. Am J Orthod and Dentofacial Orthop 2008; 133: 716-720.
 - 30) Yuasa T, Iijima M, Ito S, Muguruma T, Saito T, Mizoguchi I. Effects of long-term storage and thermocycling on bond strength of two self-etching primer adhesive systems. Eur J Orthod 2010; 32: 285-290.