

Effect of prophylactic pastes containing active ingredients on the enamel-bracket bond strength of etch-and-rinse and self-etching systems

Sarah Al-Twaijri^a; Grace Viana^b; Ana K. Bedran-Russo^c

ABSTRACT

Objective: To evaluate the effect of prophylactic pastes containing active ingredients such as amorphous calcium phosphate (ACP), fluoride, or a combination of both on the enamel-bracket tensile bond strength.

Materials and Methods: Human molars were randomly divided into four groups (N = 40) according to the type of prophylactic pastes: control (no active ingredients), paste containing fluoride, paste containing ACP, and paste containing both fluoride and ACP. After prophylaxis, 20 brackets per group were bonded using an etch-and-rinse bonding technique (37% phosphoric acid and Ortho solo), and the other remaining 20 brackets were bonded using a self-etch bonding technique (Clearfil S³Bond). Samples were tested for tensile strength 24 hours after bonding using a universal testing machine. Data were analyzed using analysis of variance (ANOVA) and Student's *t*-test with significance level at .05.

Results: The mean tensile bond strength varied between 3.043 (ACP) and 9.232 (control) MPa. The etch-and-rinse bonding technique was significantly affected by pastes containing active ingredients ($P < .001$). The self-etching system was not significantly affected by different pastes ($P > .05$).

Conclusions: Cleaning enamel surfaces with prophylactic pastes that contain ACP and/or fluoride, prior to orthodontic bonding, resulted in a statistically significant decrease in the tensile bond strength of etch-and-rinse bonding technique. (*Angle Orthod.* 2011;81:788–793.)

KEY WORDS: Enamel; Bond strength; Prophylactic paste; Etch-and-rinse systems; Self-etching system; Brackets

INTRODUCTION

When Buonocore¹ introduced the enamel etching technique to increase retention of pit and fissure resins, the orthodontic profession evolved from using bands around teeth to resin-bonded brackets attached directly onto the enamel surface. This adhesion strategy involves several steps: (1) dissolution of the

enamel hydroxyapatite using phosphoric acid to create microporosity and increase the surface bonding area, (2) application of a bonding agent composed of resin and primer, and (3) application of resin composite cement that adheres to the orthodontic bracket.²

Current bonding agents combine the enamel etching acid and the bonding agent into one acidic priming solution to minimize chair time as well as costs to the clinician and the patient.³ The phosphate group on the methacrylated phosphoric acid ester dissolves the calcium from the hydroxyapatite and is incorporated into the network when the primer polymerizes.³ Thorough cleaning with a prophylaxis instrument and paste is essential to remove not only the plaque and other surface debris, but also the enamel pellicle that acts as a barrier preventing resin penetration to the enamel surface,¹ resulting in a 50% increase in resin bond strength.^{4,5}

Orthodontic brackets and bands increase the surface area for plaque accumulation and create challenges for maintaining optimum oral hygiene. White spot lesions have been shown to appear around and

^a Orthodontist, Armed Forces Hospital, Dhahran, Saudi Arabia.

^b Instructor, Statistician, Department of Orthodontics, College of Dentistry, University of Illinois at Chicago, Chicago, Ill.

^c Assistant Professor, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, Ill.

Corresponding author: Dr Bedran-Russo, Room 551, Department of Restorative Dentistry, University of Illinois at Chicago, College of Dentistry, 801 South Paulina St, Chicago, IL 60612 (e-mail: bedran@uic.edu)

Accepted: February 2011. Submitted: October 2010.

Published Online: May 14, 2011

© 2011 by The EH Angle Education and Research Foundation, Inc.

underneath these appliances during orthodontic treatment.⁶ Therefore, it has been a goal to minimize the amount of demineralization seen with orthodontic therapy, as well as to find a new method for prevention and remineralization of the enamel surface. Fluoride is well known and is widely used for caries prevention; its incorporation into the enamel results in a surface less soluble in an acid environment.⁷ Fluoride and amorphous calcium phosphate (ACP) have been recommended to treat the enamel surface prior to cementing orthodontic bands or bonding orthodontic brackets.^{8,9} ACP can provide a sustained release of calcium and phosphate ions,¹⁰ which can reverse demineralization of enamel.^{11,12}

Bracket bond failure is a common problem in orthodontic clinics with reports of 4%–20% of bracket bond failure rate after 5-year clinical review.¹³ Replacing debonded brackets could be time-consuming for both the practitioner and the patient, adds costs to treatment, and prolongs treatment time. The effect of ACP on the shear bond strength has been reported recently when topically applied and when using ACP-containing bonding materials.^{14–17} With the introduction of new materials in the market, such as prophylactic pastes containing ACP and fluoride, it is important to understand the effect of these products on the bond strength of orthodontic brackets. The objective of this study was to test the null hypothesis that prophylactic pastes containing fluoride and/or ACP do not affect the enamel-bracket bond strength of two bonding techniques.

MATERIALS AND METHODS

Tensile Bond Strength

The use of 80 extracted human molars has been classified by the Institutional Review Board of the University of Illinois at Chicago as research that does not involve human subjects (2008-0761). The exclusion criteria included teeth with caries, teeth that visually appeared to have hypocalcifications or decalcifications, teeth with restorations, or teeth with damaged buccal or lingual enamel. The extracted teeth were kept in 0.1% thymol solution for no longer than 3 months, the roots were cut and discarded, and each crown was sagittally sectioned into buccal and lingual surfaces using a diamond wafering blade (Isomet 1000, Buehler Ltd, Lake Buff, Ill).

One-hundred sixty crown halves, which would include buccal and lingual surfaces, were mounted individually in 1 inch × 1 inch × 1 inch cylindrical acrylic blocks so that the exposed enamel surface is parallel to the base of the acrylic and facing upwards. The samples were randomly divided ($n = 20$)

according to the four prophylactic pastes and the two bonding techniques. The enamel surfaces were cleaned with the following prophylactic pastes from Premier Dental (Plymouth Meeting, Pa): control—no active ingredients (Glitter); fluoride (Glitter containing 1.23% ion fluoride); ACP (Enamel Pro); and ACP and fluoride (Enamel Pro that contains 1.23% fluoride). The enamel surfaces were cleaned using a low-speed straight handpiece and plastic prophylactic rubber cups disposed of after polishing five enamel surfaces. After cleaning the enamel surface with the designated prophylactic paste for 20 seconds, surfaces were then rinsed for 5 seconds to clear the paste from the enamel surface. A stainless steel, standard edgewise molar tube with an area of 12.74 mm² mesh base (Ormco, Glendora, Calif) was bonded to the enamel surfaces using either a self-etching system (Clearfil S³Bond, Kuraray America Co, New York, NY) or an etch-and-rinse system (Ortho solo, Ormco), both used according to manufacturers' instructions.

All brackets were bonded to the enamel surface using Transbond XT composite resin (3M Unitek, Monrovia, Calif) pushed evenly into the bracket mesh pad, and a firm pressure was placed to securely seat the bracket. Excess composite resin was removed with an explorer. The bracket was cured with an LED curing light (3M Unitek) for 20 seconds mesially and for 40 seconds occlusally. The specimens were stored in distilled water at 37°C for 24 hours prior to testing.

The tensile bond strength was evaluated using a universal testing machine (Instron, Norwood, Mass) at a crosshead speed of 1.0 mm/min. A 14-inch long, 0.016-inch wide stainless steel wire oriented perpendicular to the base was inserted into the bracket tube and attached to the upper arm of the instrument, while

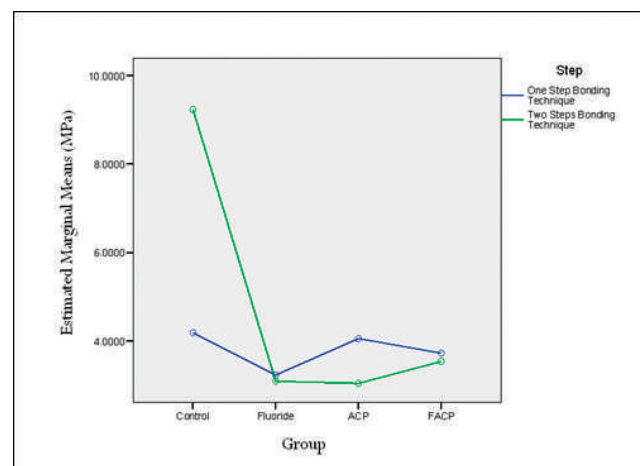


Figure 1. Profile plot of estimated marginal means of the conditions evaluated under tensile bond strength.

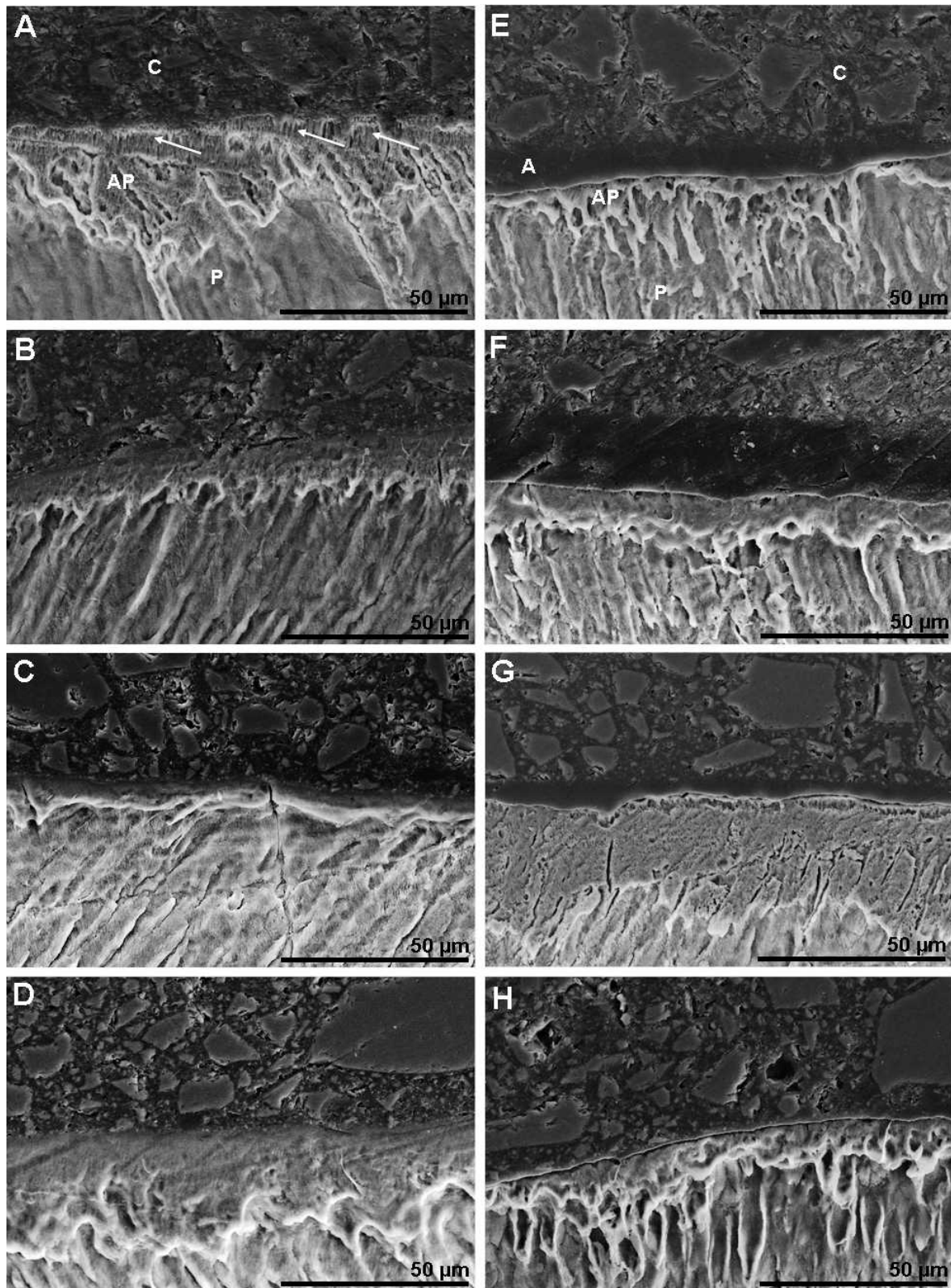


Figure 2. Representative SEM images of the bonded interface (1000 $\times$ magnification). (A) Control/Ortho solo—the enamel at the interface is irregular with the adhesive penetrating into the aprismatic layer where resin microtags can be visualized (white arrows). (C: cement; P: prismatic enamel; AP: aprismatic enamel layer). (B) Fluoride/Ortho solo—the enamel-resin interface is smooth, no resin microtags are present in enamel. (C) ACP/Ortho solo—the interface is mostly smooth with minimal irregularities. (D) ACP and fluoride/Ortho solo—the bonded interface presents similar morphology observed for fluoride and ACP groups. (E) Control/Clearfil S³Bond—a smooth enamel-resin interface indicates

Table 1. Results of the Enamel-Bracket Tensile Bond Strength of Two Bonding Techniques Following Enamel Prophylaxis With Pastes Containing Active Ingredients†

Bonding Systems	Tensile Bond Strength (MPa) – Mean (Standard Deviation)			
	Prophylactic Paste			
	No Active Ingredient (Control)	Fluoride Containing	ACP Containing	ACP and Fluoride Containing
Self-etching system (Clearfil S³Bond)	4.19 (1.65) a,A	3.23 (0.95) a,A	4.05 (2.13) a,A	3.72 (1.45) a,A
Etch-and-rinse system (Ortho solo)	9.23 (3.23) a,B	3.09 (1.46) b,A	3.04 (1.52) b,A	3.54 (2.06) b,A

† ACP indicates amorphous calcium phosphate.
* Statistical differences among groups ($P < .05$) are represented by lower and upper case letters. Different lower case letters depict statistical differences between bonding techniques for each prophylactic paste (each column). Different upper case letters depict statistical differences between prophylactic pastes for each bonding technique (each row).

the acrylic blocks holding the tooth were attached to the base.
Tensile bond strengths were calculated dividing the peak load force by the cross sectional area of the bracket meshes. Statistical analyses using analysis of variance (ANOVA) and Student's *t*-tests were performed using SPSS 16.0 software (SPSS Inc, Chicago, Ill) at a 95% confidence interval.

Interfacial Micromorphology

The cement-enamel bonded interface was evaluated using scanning electron microscopy (SEM). Samples were polished with 600–1200 grit silicon carbide, etched for 5 seconds with 10% phosphoric acid solution, rinsed, immersed in 2.5% sodium hypochlorite for 10 seconds, rinsed, dried, mounted on aluminum stub, allowed to air dry for 24 hours, and then gold sputter coated (Polaron Equipment Ltd, Watford, UK). Interfacial surfaces were examined using S3000N (Hitachi, Tokyo, Japan).

RESULTS

Two-way ANOVA showed a statistically significant interaction between the prophylactic pastes and the two orthodontic bonding techniques, ($P < .001$, Figure 1). The Student's *t*-test analysis for the tensile bond strength (MPa) between the two bonding systems showed statistically significant mean differences ($P = .029$). A one-way ANOVA of the tensile bond strength (MPa) among the four types of prophylactic pastes showed statistically significant mean differences ($P < .001$) for the two-step etch-and-rinse bonding technique. The multiple comparison

Games-Howell was performed and showed statistically significant mean differences for the control group when compared with the other three groups ($P < .001$, Table 1). There were no statistically significant differences among the four types of prophylactic pastes ($P < .05$) with the self-etching bonding technique.
Representative micrographs of the bonded interface are depicted in Figure 2. In the control group etch-and-rinse bonding technique (Ortho solo), the interface between the cement and the enamel surface is irregular as a result of the phosphoric acid application, and resin microtags can be observed. However, the cement-enamel interface of the treatment groups (active ingredients) using the same bonding technique is smoother, with lack of resin microtags, when compared with the interface of the control group (Figure 2A–D). The control group using a self-etching bonding system (Clearfil S³Bond) showed a very smooth interface, regardless of the prophylactic paste used prior to bonding (Figure 2E–H).

DISCUSSION

The incorporation of active ingredients to products used during the orthodontic bracket bonding technique could alter the bracket bond strength to enamel depending on the bracket bonding system.¹⁸ The present study shows that prophylactic pastes containing active ingredients have a detrimental effect on the enamel-bracket bond strength when using an etch-and-rinse system. Previous studies have reported similar results with pit and fissure sealants after using topical fluorides which have shown decreased enamel solubility, resulting in decreased bond strength.^{19,20} Clinically, the etching of enamel creates microporos-

← minimum enamel etching pattern provided by the bonding technique. In addition, a uniform adhesive layer (A) is observed due to light curing of the bonding system prior to cementation. (C: cement; P: prismatic enamel; AP: aprismatic enamel layer; A: adhesive layer). (F) Fluoride/Clearfil S³Bond—the adhesive interface is very smooth and the adhesive layer appears to be uniform in thickness. (G) ACP/Clearfil S³Bond—the adhesive interface is very smooth and resembles the control group. (H) ACP and fluoride/Clearfil S³Bond—similar morphology of that observed for control, fluoride, and ACP groups.

ities within the enamel and reduces surface tension that allows the resin to penetrate and polymerize within the etched enamel rods.²¹ Teeth with higher concentration of fluoride are more resistant to acid etching than normal teeth due to the formation of fluorapatite in the structure of the enamel surface, and topical fluoride application adversely affects etching treatment.²¹ In addition, fluoride results in the formation of reaction products. The reaction products such as $\text{Ca}_5(\text{PO}_4)_3\text{F}$ and CaF_2 are formed after sodium fluoride (NaF) treatment, and $\text{Sn}_3\text{PO}_4\text{F}_3$ and CaF_2 are formed after stannous fluoride (SnF_2) treatment. These products partly or completely fill the interprismatic space and result in a smaller bonding area as well.²² Therefore, the decrease in tensile bond strength for the fluoride prophylactic paste group is most likely due to decreased enamel solubility after the incorporation of the fluoride ions onto the enamel surface. The smooth cement-enamel interface observed in the SEM may indicate reduced retention and mechanical interlocking (Figure 2B).

Prophylactic paste containing ACP resulted in lower bond strength when using the etch-and-rinse bonding technique as well. ACP has been added to many dental products and has been shown *in vitro* to be effective as treatment of early caries lesions²³ and as an adjunct for children with challenged oral hygiene habits.²⁴ The products work by providing calcium and phosphate ions, which are essential in the formation of hydroxyapatite, and in the presence of fluoride, hydroxy-fluorapatite. Contrary to our findings, significant increased shear bond strength was reported after application of casein phosphopeptide (CPP)-ACP.¹⁴ The conflicting results may be explained by (1) the use of human teeth since bovine enamel presents larger crystal grains and more lattice defects,²⁵ which may affect the interaction with active ingredients; (2) evaluation of specimens immediately as opposed to 6 weeks following bonding¹⁴; and (3) tensile forces as opposed to shear forces. While tensile and shear testing are acceptable methods of evaluation, Powers et al.²⁶ have indicated that pure shear is difficult to achieve since it incorporates elements of torsion and tension. We believe the lower tensile bond strength measurement observed with the enamel surfaces treated with the ACP prophylactic pastes is also due to the increased enamel resistance to the acid-etching process. The same can be extrapolated when using the prophylactic paste that contains ACP and fluoride; combining fluoride ions with amorphous calcium phosphate resulted in similar, low tensile bond strength as compared with the groups that received only ACP or only fluoride. The smooth cement-enamel interface seen in the SEM images obtained from the surfaces that were treated with prophylactic pastes containing

ACP or ACP and fluoride supports the tensile bond strength findings (Figure 2C,D,G,H).

The results have demonstrated no statistically significant differences between the different pastes when using a self-etching adhesive. Previous studies observed significantly lower enamel-bracket shear bond strength of four different self-etching adhesive groups when compared with a total etch system.²⁷ The etching pattern of self-etching systems is shallow, which could be due to a poorer penetration of the primer into enamel porosities or the result of interference from calcium precipitates on the enamel surface, which limits further dissolution of the apatite and therefore limits the depth of demineralization.²⁸ Bonding the enamel surface with Clearfil S³Bond resulted in a similar cement-enamel interface appearance whether the prophylactic pastes contained active ingredients or not (Figure 2). The mechanism of adhesion of Clearfil S³Bond to enamel is based on the presence of 10-methacryloxydecyl dehydrogenate phosphate (10-MDP) monomer.²⁹ This functional phosphate monomer chemically adheres to hydroxyapatite, and this adhesion is reported to be very stable³⁰; thus, the system may not rely as much on the etching pattern for mechanical interlocking, but rather on the chemical adhesion to hydroxyapatite. The presence of fluoride, ACP, or both on the enamel surface would affect the etch-and-rinse bond technique that depends on the enamel roughness for adhesion; however, these products did not affect the possible chemical interaction of the Clearfil S³Bond to the enamel surface. There are no instructions on bracket cementation for Clearfil S³Bond. Therefore, restorative instructions were followed and may have affected the bond strength values, but most likely have not affected the differences between prophylactic pastes. Clearfil S³Bond may have limited clinical use in orthodontics due to the low bond strength, regardless of the prophylactic pastes.

Therefore, use of prophylactic pastes that contain active ingredients such as fluoride and ACP may not be used prior to the bonding process when using Ortho solo. The use of such products that are targeted towards the remineralizing and strengthening of the enamel surface are encouraged to be used only after the bonding process is complete. The null hypothesis was rejected.

CONCLUSIONS

- Cleaning enamel surfaces with prophylactic pastes containing ACP and/or fluoride prior to orthodontic bonding decreases the enamel-bracket tensile bond strength of an etch-and-rinse bonding system (Ortho solo).

- A self-etching bonding system (Clearfil S³Bond) was not affected by the presence of the active ingredients present in the prophylactic pastes.

ACKNOWLEDGMENT

We would like to thank Premier Dental, Ormco, and Kuraray for supplying the materials.

REFERENCES

- Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res*. 1955;34:849–853.
- Van Meerbeek B. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent*. 2003;28:215–235.
- Buyukilmaz T, Usumez S, Karaman AI. Effect of self-etching primers on bond strength—are they reliable? *Angle Orthod*. 2003;73:64–70.
- Miura F, Nakagawa K, Ishizaki A. Scanning electron microscopic studies on the direct bonding system. *Bull Tokyo Med Dent Univ*. 1973;20:245–260.
- Gultz J, Kaim J, Sherer W. Treating enamel surfaces with a prepared pumice prophyl paste prior to bonding. *Gen Dent*. 1999;47:200–201.
- Ogaard B, Rølla G, Arends J. Orthodontic appliances and enamel demineralization. Part 1. Lesion development. *Am J Orthod Dentofacial Orthop*. 1988;94:68–73.
- ten Cate JM. Current concepts on the theories of the mechanism of action of fluoride. *Acta Odontol Scand*. 1999;57:325–329.
- Ogaard B, Rølla G, Arends J, ten Cate JM. Orthodontic appliances and enamel demineralization. Part 2. Prevention and treatment of lesions. *Am J Orthod Dentofacial Orthop*. 1988;94:123–128.
- Uysal T, Amasyali M, Koyuturk AE, Ozcan S. Effects of different topical agents on enamel demineralization around orthodontic brackets: an in vivo and in vitro study. *Aust Dent J*. 2010;55:268–274.
- Park MS, Eanes ED, Antonucci JM, Skrtic D. Mechanical properties of bioactive amorphous calcium phosphate/methacrylate composites. *Dent Mater*. 1988;14:137–141.
- Tung MS, Eichmiller FC. Dental applications of amorphous calcium phosphates. *J Clin Dent*. 1999;10(1 Spec No):1–6.
- Skrtic D, Hailer AW, Takagi S, Antonucci JM, Eanes ED. Quantitative assessment of the efficacy of amorphous calcium phosphate/methacrylate composites in remineralizing caries-like lesions artificially produced in bovine enamel. *J Dent Res*. 1996;75:1679–1686.
- Mizrahi E. Orthodontic bands and directly bonded brackets: a review of clinical failure rate. *J Dent*. 1983;11:231–236.
- Keçik D, Cehreli SB, Sar C, Unver B. Effect of acidulated phosphate fluoride and casein phosphopeptide-amorphous calcium phosphate application on shear bond strength of orthodontic brackets. *Angle Orthod*. 2008;78:129–133.
- Foster JA, Berzins DW, Bradley TG. Bond strength of an amorphous calcium phosphate-containing orthodontic adhesive. *Angle Orthod*. 2008;78:339–344.
- Xiaojun D, Jing L, Xuehua G, Hong R, Youcheng Y, Zhangyu G, Sun J. Effects of CPP-ACP paste on the shear bond strength of orthodontic brackets. *Angle Orthod*. 2009;79:945–950.
- 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.
- Grajower R, Glick A, Gedalia I, Kochavi D. Tensile strength of the bond between resin to enamel etched with phosphoric acid containing fluoride. *J Oral Rehabil*. 1979;6:267–272.
- Lee H, Stoffey D, Orlowski J, Swartz ML, Ocumpaugh D, Neville K. Sealing of developmental pits and fissures. 3. Effects of fluoride on adhesion of rigid and flexible sealers. *J Dent Res*. 1972;51:191–201.
- Low T, Von Fraunhofer JA, Winter GB. Influence of the topical application of fluoride on the in vitro adhesion of fissure sealants. *J Dent Res*. 1977;56:17–20.
- Brantley WA, Eliades T. *Orthodontic Materials: Scientific and Clinical Aspects*. New York, NY: Thieme; 2001.
- Kochavi D, Gedalia I, Anaise J. Effect of conditioning with fluoride and phosphoric acid on enamel surfaces as evaluated by scanning electron microscopy and fluoride incorporation. *J Dent Res*. 1975;54:304–309.
- Kumar VL, Ilthagarun A, King NM. The effect of casein phosphopeptide-amorphous calcium phosphate on remineralization of artificial caries-like lesions: an in vitro study. *Aust Dent J*. 2008;53:34–40.
- Reynolds EC. Calcium phosphate-based remineralization systems: scientific evidence? *Aust Dent J*. 2008;53:268–273.
- Moriwaki Y, Kani T, Kozatani T, Tsutsumi S, Shimode N, Yamaga R. The crystallinity change of bovine enamel during maturation. *Jpn J Dent Mat*. 1968;9:78–85.
- Powers JM, Kim H, Turner DS. Orthodontics adhesives and bond strength testing. *Semin Orthod*. 1997;3:147.
- 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.
- Davari AR, Yassaei S, Daneshkazemi AR, Yosefi MH. Effect of different types of enamel conditioners on the bond strength of orthodontic brackets. *J Contemp Dent Pract*. 2007;1:36–43.
- Hegde MN, Bhandary S. An evaluation and comparison of shear bond strength of composite resin to dentin, using newer dentin bonding agents. *J Conserv Dent*. 2008;11:71–75.
- Yoshida Y, Nagakane K, Fukuda R, et al. Comparative study on adhesive performance of functional monomers. *J Dent Res*. 2004;83:454–458.