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Effect of a New Surface Treatment Solution on the Bond Strength of Composite to Enamel

ABSTRACT

Clean & Boost (Apex Dental Materials) is a novel surface treatment solution designed to be used in place of phosphoric acid to increase the bond strength of self-etch adhesives to enamel and more effectively remove contaminants (e.g., handpiece lubricant) from the tooth surface. Simplicity (Apex Dental Materials) is a self-etch bonding agent with acidic monomers that reportedly eliminates concerns about bonding to enamel. The purpose of this study was to evaluate the effect of surface exposure, surface treatment, or bonding agent on the bond strength of composite to enamel. The crowns of 120 bovine incisors were mounted. For surface exposure, half of the specimens were exposed to the water spray of a high-speed handpiece after cleaning/lubrication with an automatic handpiece system and the other half had only water spray. For surface treatment, one third of the specimens were etched with 34% phosphoric-acid gel, one third were treated with Clean & Boost, and one third were untreated. Self-etch bonding agents (Simplicity or Clearfil SE Bond, Kuraray) were applied and light cured. Composite was placed into a mold and light cured (n=10). The specimens were stored in water for 24 hours at 37°C and tested in shear. Data were analyzed with a 3-way ANOVA/Tukey's to evaluate the effects of surface exposure, surface treatment, or bonding agent on the bond strength of composite to enamel (alpha=0.05). A significant difference was found based on surface treatment ($p=0.023$), or bonding agent ($p<0.001$), but not on surface exposure ($p=0.057$) with no significant interactions ($p>0.21$). Phosphoric-acid etch resulted in greater bond strength than no surface treatment. The use of Clean & Boost was not significantly different from phosphoric acid or no treatment. Simplicity had significantly lower bond strengths to enamel than Clearfil SE Bond. Handpiece-spray exposure had no significant effect on bond strength to specimens.

Clinical Implications: The novel surface treatment solution (Clean and Boost) or the strongly acidic self-etch bonding agent (Simplicity) did not provide any advantages in bonding composite to enamel compared to the traditional use of phosphoric acid or the milder self-etch bonding agent (Clearfil SE Bond) with or without the exposure of enamel to the spray of a lubricated handpiece.

INTRODUCTION

Adhesive bonding agents were first introduced to dentistry in the 1950's along with composite-resin restorative materials (Meharry et al., 2013). Since then, seven generations of adhesives has been introduced. Adhesive bonding agents consist of three basic components (i.e., conditioner, primer, bonding resin) in multiple combinations to create the various generations. Adhesive bonding agents may be classified as either "etch-and-rinse" or "self-etch" systems (Hilton et al., 2013).

Dentistry adapted the etch-and-rinse philosophy ever since Buonocore increased the resin-enamel bond strength by 100-fold via etching of enamel with phosphoric acid (Buonocore, 1955). The etch-and-rinse approach relies on a strong acid (pH 0.1-0.4) to demineralize both dentin and enamel. For dentin, the stronger acid demineralizes the intertubular dentin and creates micro-meter-sized porosities within the underlying collagen fibrillar matrix. For enamel, the stronger acid creates a 5-50 µm thick microporous layer (Hilton et al., 2013). These porosities endorse the bonding resin to infiltrate and produce an interlocking hybrid layer or interdiffusion zone. The stronger acid must be removed by rinsing, hence the name "etch-and-rinse" (Pashley et al., 2011).

The self-etch approach, on the other hand, does not require the etchant to be rinsed off the surface. Etchant and primer are the same chemical system, utilizing acidic monomers capable of etching and priming at the same time. The acidity of the acidified primer varies between strong (pH < 1), mild (pH ~ 2), or ultramild (pH ~2.5). The mild and ultramild acidified primers only partially demineralize dentin up to 1µm (Hilton et al., 2013). As a result, the collagen fibrils are protected by residual hydroxyapatite crystals. In addition, certain acidic monomers (e.g., 10-methacryloyloxydecyl dihydrogen phosphate or 10-MDP) are able to form stable calcium-phosphate and calcium-carboxylate covalent bonds with residual hydroxyapatite (Van Meerbeek et al., 2011). Hence the mild or ultramild self-etch approach may have both mechanical and chemical retention. The strong acidified primers perform similar to the etch-and-rinse approach, in terms of complete removal of smear layer and demineralization of superficial hydroxyapatite, to create a several micron thick scaffold of the porous layer for resin to infiltrate micromechanically (Hilton et al., 2013).

The etch-and-rinse approach is available in three- or two-step systems. In the three-step system, all three components of the bonding agent are applied separately. To simplify the process, manufacturers combined the primer and resin components to create a two-step system. Self-etch adhesive bonding agents were more recently introduced and are divided into two- and one-step systems. Two-step self-etch adhesive agents combine the acidic conditioner with the primer in the initial step and use a bonding resin in the second step. Even further reduction in the number of steps came with the introduction of one-step, self-etch adhesives with the acidified primer and bonding resin placed in one simplified step (Van Meerbeek et al., 2011).

A meta-analysis of controlled clinical studies was conducted by Heintze et al. in 2010 that examined the retention rates of cervical composite restorations bonded with various adhesive agents. As a result of the review, it was concluded that the highest retention rates were achieved with the mildly acidic two-step, self-etch adhesive, Clearfil SE Bond (Kuraray, New York, NY), followed closely by the three-step, etch-and-rinse adhesive Optibond FL (Kerr, Orange, CA) (Heintze et al., 2010). In a recent 2014 systematic review of the literature, Peumans et al. also reported the average annual failure rate of non-carious cervical lesions bonded with different dental adhesives and restored with composite resin. The mild two-step self-etch adhesives, such as Clearfil SE Bond, were once again found to be the most effective clinically with an annual failure rate of 2.5%, followed closely by the three-step etch-and-rinse adhesives, such as Optibond FL, at 3.1% (Peumans et al., 2014).

With the evolution of self-etch adhesive systems, there was a concern that the manufacturers were sacrificing the strength of the bond to enamel by using a weaker acidified primer in order to eliminate one step in the procedure. Enamel bonding primarily occurs by the micromechanical interlocking of adhesive resin into microporosities of the etched surface. Laboratory studies have demonstrated that self-etch adhesives produce lower bond strength to enamel, and particularly uncut enamel (Erickson et al., 2005). More significantly, clinical studies have shown significantly less marginal defects and staining with selective etching of enamel with phosphoric acid when using a self-etch adhesive agent (Peumans et al., 2007; Peumans et al., 2010; Ermis et al., 2010). Although no significant difference in retention rates in restored non-carious lesions with or without the selective etching of enamel were found in the systematic review by Peumans and others, a recent article by Van Meerbeek and Yoshida state that the use of phosphoric acid on enamel currently remains necessary to maintain the durable bond to the interface and to reduce marginal degradation and staining (Peumans et al., 2014; Van Meerbeek and Yoshida, 2014)

Simplicity (Apex Dental Materials, Lake Zurich, IL) is a two-step self-etch adhesive with more strongly acidic monomers that reportedly produces etching results identical to a phosphoric-acid gel. According to the manufacturer, Simplicity eliminates concerns about bonding to enamel with self-etch adhesives to ensure long-term margin integrity (www.apexdentalmaterials.com). Very little research has been published evaluating this more strongly acidic self-adhesive bonding agent. Tay et al. (2004), found no significant difference in the thickness of the hybrid layer and the microtensile bond strength to unground enamel between Simplicity and an etch-and-rinse adhesive.

Apex Dental Materials has introduced an aqueous cleanser called Clean and Boost which is designed to be used in place of phosphoric acid to etch enamel and dentin and to more effectively remove contaminants such as tooth debris, temporary cement, bacteria, blood, saliva, handpiece oil, and imaging powder from the tooth surface. According to the Material Data Safety Sheet, Clean and Boost contains a proprietary blend of organic and inorganic acids and a low viscosity hydrophilic monomer (hydroxyethyl methacrylate). Along with cleansing the surface, Clean & Boost will reportedly increase the bond strengths of self-etch adhesives and cements to enamel to prevent microleakage, staining, and accelerated wear of the margins (www.apexdentalmaterials.com). No research has been accomplished evaluating this new surface cleanser.

Laboratory studies have evaluated the effect of various types of contaminants on adhesive bonding to enamel and dentin. Some studies have evaluated the effect of various handpiece maintenance sprays on the bond strength of composite to enamel and dentin. However, the results of laboratory research have been equivocal, with some studies finding no effect and others finding a loss in bond strength (Sugawara et al., 2010; Matos et al., 2008; Xie et al., 1993; Powers et al., 1995; Rosa et al., 2000; Knight et al., 1999; Roberts et al., 2005).

The purpose of this study was to evaluate the effect of type of surface exposure (none or handpiece spray), surface treatment (none, phosphoric acid, or Clean & Boost) or bonding agent (Simplicity or Clearfil SE Bond) on the shear bond strength of a composite restorative material to enamel. The null hypotheses to be tested were that there would be no difference in the shear bond strength of composite to bovine enamel based on 1) surface exposure, 2) surface treatment, or 3) bonding agent.

MATERIALS AND METHODS

One hundred and twenty extracted bovine incisors were purchased and stored in 0.5% chloramine-T (Science Stuff, Austin, TX). The teeth were sectioned buccolingualy at the cemento-enamel junction to remove the root using a distilled water-cooled diamond saw (Isomet Slow-Speed Saw, Buehler, Lake Bluff, IL). Retention cuts were placed in the lingual surface of the crown to prevent tooth dislodgement during shear testing. The teeth were mounted in polyvinylchloride (PVC) pipe using dental stone. To facilitate bonding, a small flattened area was ground on the facial surface using a diamond wheel (#818, Brasseler, Savannah, GA) in a drill press (TBM 115, Proxxon, Hickory, NC).

The enamel specimens were divided into twelve groups with ten specimens each as depicted in Table 1. For surface exposure, half of the specimens were exposed to the lubricant of a highspeed dental handpiece and the other half of the specimens were rinsed with water only. The Midwest Tradition highspeed handpiece (Dentsply, York, PA) requires lubrication after each use. Five handpieces were automatically lubricated by NSK Care 3 Plus according the manufacture instruction. The handpieces were sterilized using a steam autoclave (V-120 Prevac Steam Sterilizer, Steris, Mentor, OH) according to the specifications of the manufacturer. Prepared enamel surfaces were then exposed to 30 seconds of spray from the handpiece approximately 10 mm from the enamel surface with a bur blank (Dentsply) operating at full speed using a vinyl polysiloxane jig. See Figure 1.

<table>
<tr><th></th><th>Surface Exposure</th><th>Surface Treatments</th><th>Bonding Agents</th><th>Composite</th></tr>
<tr><td>1.</td><td>Handpiece spray</td><td rowspan="2">Phosphoric Acid</td><td rowspan="6">Clearfil SE Bond</td><td rowspan="12">Filtek Z250</td></tr>
<tr><td>2.</td><td>Water Spray</td></tr>
<tr><td>3.</td><td>Handpiece spray</td><td rowspan="2">Clean & Boost</td></tr>
<tr><td>4.</td><td>Water Spray</td></tr>
<tr><td>5.</td><td>Handpiece spray</td><td rowspan="2">No Treatment</td></tr>
<tr><td>6.</td><td>Water Spray</td></tr>
<tr><td>7.</td><td>Handpiece spray</td><td rowspan="2">Phosphoric Acid</td><td rowspan="6">Simplicity</td></tr>
<tr><td>8.</td><td>Water Spray</td></tr>
<tr><td>9.</td><td>Handpiece spray</td><td rowspan="2">Clean & Boost</td></tr>
<tr><td>10.</td><td>Water Spray</td></tr>
<tr><td>11.</td><td>Handpiece spray</td><td rowspan="2">No Treatment</td></tr>
<tr><td>12.</td><td>Water Spray</td></tr>
</table>

Table 1. Treatment Groups

Figure 1: Enamel specimen exposed to handpiece spray

For surface treatment, one third of the specimens were etched with 34% phosphoric-acid gel etchant (Dentsply, York, PA) for 15 seconds, rinsed with water for 15 seconds, then lightly air dried for three seconds. One third of the specimens were treated with Clean & Boost according to manufacturer's instructions (Table 2). The final one third of the specimens were untreated. The bonding agents (Clearfil SE Bond or Simplicity) were applied according to the manufacturer's instruction (Table 2) and light cured with a visible light-curing unit (Bluephase G2, Ivoclar Vivadent, Amherst, NY). Irradiance was determined with a radiometer (Bluephase Meter, Ivoclar Vivadent) and was considered acceptable if greater than 1000 mW/cm^2.

Products	Applications	Composition	Manufacturer Instruction
Clearfil SE Bond	Primer	Hydroxyethyl Methacrylate Methacryloyloxydecl Hydrophobic aliphatic dimethacrylate Camphorquinone Water Accelerators Dyes Others	* Apply for 20 s on tooth surface * Evaporate volatile ingredients with a mild air stream
	Bonding Adhesive	Bisphenol A glycidyl methacrylate Hydroxythyl methacrylate Methacryloyloxydecl dihydrogen phosphate Hydrophobic aliphatic dimethacrylate Collodal silica Initiators Accelerators Others	* Apply to tooth surface * Expose to air stream * Light cure for 10 s
Simplicity	Primer	Blend of organic and inorganic acid	* Apply on tooth surface and agitating briskly for 10 s * Do not air dry
	Bonding Adhesive	Hydroxyethyl methacrylate Acetone	* Apply 3 layers to tooth surface and air dry for 10 s * Apply a 2 layers and air dry for 5 s * Light cure for 10 s
Clean & Boost	Surface Treatment	Blend of organic and inorganic acid 2-Hydroxyethyl methacrylate	* Apply on tooth surface and agitating briskly for 10 s * Rinse thoroughly

Filtek Z250	Composite Resin	Bisphenol A polyethylene glycol dimethacrylate Diurethane dimethacrylate Bisphenol A diglycidyl ether dimethacrylate Triethylene glycl dimethacrylate Initiators Zirconia, Silica	*Place Z250 resin in increments less than 2.5mm thick *Light cure each increment for 20 s

Table 2: Product Composition and Instructions

After application of the adhesive, each bonded specimen was placed in a jig (Ultradent Products, South Jordan, UT) and secured beneath a white plastic mold. The bonded area was limited to the 2.4 mm circle determined by the mold. The composite resin restorative material (Filtek Z250, St. Paul, MN) was incrementally placed to a height of 3-4 mm. Each layer was polymerized as recommended by the manufacturer with the visible curing light unit. The specimens were stored for 24 hours in distilled water at 37˚C in a laboratory oven (Model 20GC, Quincy Lab, Chicago, IL).

The bond strength was tested in shear mode with a knife-edge probe in a universal testing machine (Model 5943, Instron, Norwood, MA) at a crosshead speed of 1 mm/minute until failure. Shear bond strength values in megapascals (MPa) were calculated in newtons from the peak load of failure divided by the specimen cross-sectional surface area. The mean and standard deviation was determined per group. Data were analyzed with a 3-way ANOVA and Tukey's post hoc tests to evaluate the effects of surface exposure, surface treatment, or bonding agent on the shear bond strength of composite to bovine enamel. Following testing, the specimens were examined under the 10x microscope to determine the failure mode as either: 1) adhesive fracture at the adhesive interface, 2) cohesive fracture in the enamel or composite, or 3) mixed (combination of adhesive and cohesive) in enamel or composite.

RESULTS

Significant differences were not found in shear bond strength based on surface exposure ($p=0.057$). Handpiece spray exposure had no significant effect on bond strength to enamel. Significant differences were found based on surface treatment ($p=0.023$). Phosphoric-acid etch resulted in greater bond strength overall (19.7 ± 8.9 MPa) than no surface treatment (16.0 ± 7.7 MPa). The use of Clean & Boost (18.1 ± 8.4 MPa) was not significantly different from phosphoric acid or no surface treatment. Significant differences were found based on bonding agent (p<0.001). Clearfil SE Bond (23.7 ± 7.4 MPa) had significantly greater bond strengths to enamel overall compared to Simplicity (12.2 ± 4.5 MPa). See Table 3. There was no significant interactions among the groups using the 3-way ANOVA (p>0.21). Less adhesive failures were seen with the use of Clearfil SE Bond or phosphoric acid. See Figure 2.

Bonding Agent	**Shear Bond Strength** MPa (st dev)						
	No Treatment		**Clean & Boost**		**Phosphoric Acid**		
	Handpiece Spray	Water Spray	Handpiece Spray	Water Spray	Handpiece Spray	Water Spray	Total Mean
Simplicity	10.6 (4.0)	11.4 (4.3)	11.4 (4.4)	14.5 (4.4)	11.7 (2.1)	13.4 (5.4)	12.2 (4.5) A
Clearfil SE	19.2 (8.6)	22.9 (3.5)	20.8 (6.4)	25.8 (8.4)	27.9 (4.3)	25.7 (7.1)	23.7 (7.4) B
Total Mean	16.0 (7.7) a		18.1 (8.4) ab		19.7 (8.9) bc		
Groups with the same lower case letter per row or upper case letter per column are not significantly different (p>0.05)							

Table 3. Shear Bond Strength

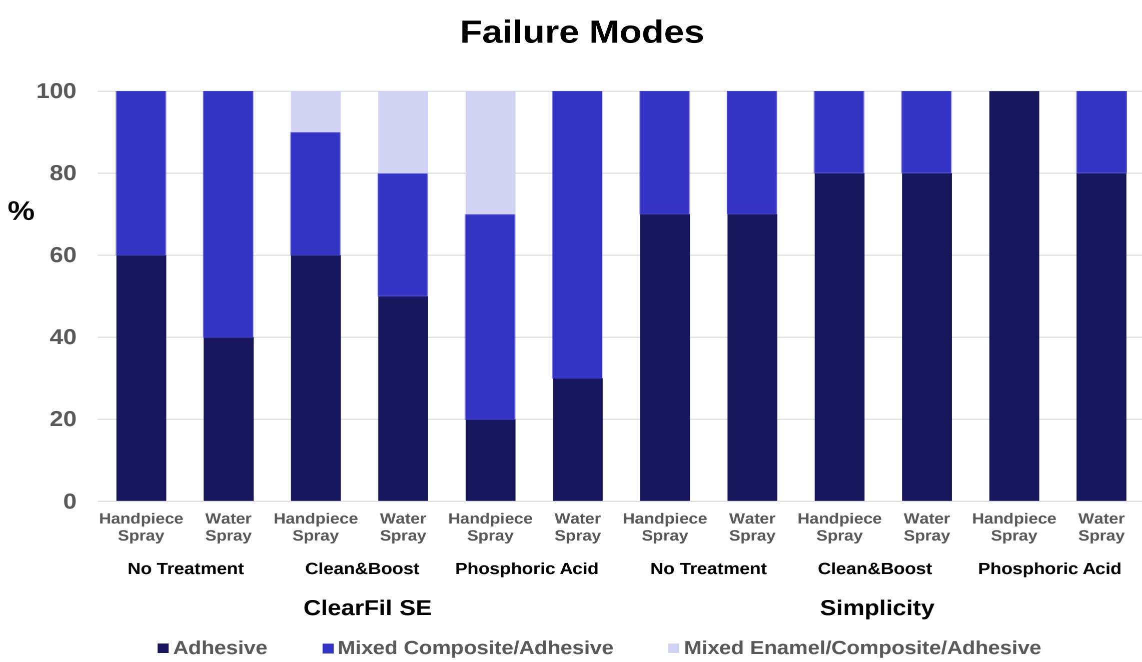

Figure 2. Failure Modes

DISCUSSION

A new surface treatment solution, Clean & Boost, was designed to be used in place of phosphoric acid for the purpose of cleaning and disinfecting freshly prepared tooth surface. No research has been published evaluating this new surface treatment product against the gold standard, phosphoric acid. Simplicity is a self-etch bonding agent with acidic monomers with lower pH to reportedly provide better bonding to enamel. Very limited research is available evaluating this acidic self-etch bonding agent.

The first null hypothesis that surface exposure had no effect on shear bond strength was not rejected. There was no difference in shear bond strength of composite to enamel with or without exposure to the spray of a lubricated highspeed handpiece. In addition to handpiece lubricant, contaminants such as saliva, blood, dental cements, and imaging powder could have an effect on bonding of composite to the tooth structure. Handpiece lubricant was chosen to be the contaminant for this study due to its hydrophobic nature, and clinically, it may be more difficult to prevent the exposure of teeth from handpiece lubricant compared to blood or saliva. One study has demonstrated that a sterilized handpiece could discharge residual lubricant for at least 4 hours (Pong et al., 2005). A number of studies have examined the effect of handpiece lubricant on the shear bond strength of composite to enamel or dentin (Sugawara et al., 2010; Matos et al., 2008; Xie et al., 1993; Powers et al., 1995; Rosa et al., 2000; Knight et al., 1999; Roberts et al., 2005). The studies found minimal or no effect of handpiece lubricant on the bond strength to enamel, but the effect on dentin was found to be more significant.

A study by Sugawara et al. (2010) showed a significant effect of handpiece lubricant on shear bond strength to dentin. Matos et al. (2008) evaluated the effect of handpiece lubricant on both dentin and enamel bond strengths and found reduced bond strengths, but the effects were less detrimental on enamel compared to dentin. Two studies with the same group of researchers (Xie et al., 1993 and Powers et al., 1995) evaluated the effect of multiple contaminants (including handpiece lubricant) on the both dentin and enamel bond strengths. Xie et al., found that the effects of handpiece lubricant was somewhat equivocal. On both enamel and dentin surfaces, bond strengths increased for one bonding agent but decreased for another. Powers et al., found that the handpiece lubricant significantly reduced the bond strengths to dentin but not to enamel. Rosa et al., (2000) found that handpiece oil had no significant effect on bonding to enamel. However, in all of these studies, the handpiece oil was applied directly to the surfaces.

Two studies evaluated the effect of the spray from a lubricated handpiece to simulate a clinical scenario. When bonding to enamel, Knight et al. (1999) found that the spray from a lubricated handpiece significantly reduced the bond strength if the handpiece was not purged for thirty seconds immediately before spraying. This differs from the results of our study which showed no significant reduction in bond strength using a handpiece that was not purged before spraying. Although there was a trend for the spray of the handpiece to result in lower bond strengths in this study, it did not result in a statistically significant difference from water spray alone (p=0.057). When bonding to dentin, a study by Roberts et al. (2005) found no significant effect on bond strength with or without exposure of the dentin surface to the spray from a lubricated handpiece that was purged for thirty seconds before spraying.

The second null hypothesis was rejected. Differences in shear bond strength were found based on the type of surface treatment. Phosphoric-acid etch resulted in greater bond strength overall than no surface treatment. Phosphoric acid has been extensively researched ever since 1955 when Buonocore published his studies evaluating the concentration and the timing of etching, rinsing, and drying (Summitt et al., 1992). No research has been published evaluating the bond strength of composite to tooth structure using Clean and Boost. In this study, Clean & Boost did not provide a significant increase in bond strength of the self-etch bonding agent compared to no surface treatment. Clean and Boost contains a proprietary blend of organic and inorganic acids and a low viscosity hydrophilic monomer (hydroxyethyl methacrylate).

The third null hypothesis was also rejected. Differences were found in bond strength based on the type of bonding agent. Clearfil SE Bond had significantly greater bond strengths to enamel overall compared to Simplicity. In addition Clearfil SE Bond had less adhesive failures than Simplicity, which suggests a stronger bonding interface between the adhesive and enamel (Al-Salehi and Burke, 1997). Both Clearfil SE and Simplicity are two-step self-etching bonding agents, but with different pH levels. Clearfil SE Bond is an ultra-mild self-etch system and Simplicity is a strong self-etch system. A strong self-etch adhesive ideally should etch enamel to produce a similar result as phosphoric acid, which leaves a porous enamel surface. (Hilton, 2013). Tay et al., (2004) found that strongly acidic self-etch bonding agents achieved similar bond strengths to unground enamel using phosphoric acid, however, the thickness of the enamel hybrid layer was less. The degree of demineralization produced by self-etch adhesives depends largely on the acidity or etching aggressiveness of the functional monomer and is material dependent. According to Sunfield et al., (2005) the penetration of the adhesive system may be restricted to the more superficial enamel layers with creation of shorter resin tags when self-etch adhesives are used without a selective-etch step using phosphoric acid. Erickson et al., (2009) also found improved bond strengths with a selective-etch step and attributed this to the degree of etching or the etch morphology achieved. The enamel hybrid layer is very important in etch-and-rinse adhesive systems because it allows resin to penetrate and form a mechanical interlocking interface (Hilton, 2013). The ultra-mild self-etch system in Clearfil SE Bond does not demineralize tooth surface like phosphoric acid. In a study by Tay et al., (2004), the weaker acidic self-etch adhesive (Clearfil SE Bond) only achieved a fine pitting of the enamel surface and corresponding fine resin projections. However, neither the acidity of the adhesive agent, thickness of the hybrid layer, nor the length of the resin tags are solely responsible for bonding effectiveness and stability of adhesives. Clearfil SE Bond relies on its functional acidic monomer, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), to form a chemical bonding with hydroxyapatite (Van Meerbeek et al., 2011). The monomer, 10-MDP contains phosphate groups, capable of producing chemical bonds with calcium in hydroxyapatite (Moszner et al., 2005). Although the bond strengths to enamel were improved with phosphoric-acid etching, this study confirmed previous studies that demonstrated that an ultra-mild self-etch adhesive, Clearfil SE Bond, was capable of achieving strong bonds to ground enamel even without a selective-etch step (DeMunck et al., 2005; McLean et al., 2015).

CONCLUSIONS

The more strongly acidic two-step self-etch bonding agent (Simplicity) had significant lower bond strengths to ground enamel than the mildly acidic two-step self-etch bonding agent (Clearfil SE Bond). A phosphoric-acid etch resulted in greater bond strength than no surface treatment. Less adhesive failures were seen with the use of Clearfil SE Bond and phosphoric acid. The use of the aqueous cleanser (Clean & Boost) was not significantly different from phosphoric acid or no treatment. Handpiece-spray exposure had no significant effect on bond strength to enamel.

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