INTRODUCTION

Phosphoric acid (PA) etching has been employed to improve enamel bond strength since Buonocore’s report was published1). Furthermore, through the development of bonding systems containing hydrophilic resin monomers to enhance their coupling with wet dentin substrates, the simultaneous bonding application of enamel to dentin has been achieved 2,3). Etch&Rinse systems involve, in their most conventional form, three steps with successive applications of the acid etchant, followed by that of the primer, and, eventually, by the application of the actual bonding agent or adhesive resin4,5). The primer functioned as an adhesion promoter for dentin, not for enamel 5). Subsequently, self-etch adhesives (SEAs) that afforded an acidic self-etched primer were developed by combining the acid etchant with a primer5).

Recently, a two-step self-etching adhesives (2-SEAs) and one-bottle SEAs (1-SEAs) have been widely accepted in clinical situations6,7). However, there are some indications that they do not demineralize the enamel sufficiently, although 1-SEAs are reported to be clinically acceptable. The clinical study reported that marginal discolorations occurred at the enamel interface over time7). To overcome this drawback, clinicians sometimes apply PA to enamel prior to 1-SEA8,9).

The quality of an enamel bond can be influenced by the pH of the adhesive as well as the composition of the bonding systems and the method of applying their components to the enamel10,11). Some studies have focused on the influence of the dentin primer on enamel bond strength12-14). Moreover, the application of the hydrophilic dentin primers tends to reduce the enamel bond strength12,13,15). However, the dentin primers do not generally cause a reduction in the bond strength of pre-etched enamel14).

Nevertheless, the morphological evaluation of the bonding interface and the bond strength are also crucial. To assess the resistance of the bonding interface to secondary caries, some studies performed the interfacial observation after the attack of the artificial demineralizing solution and reported the formation of the acid–base resistant zone (ABRZ)16). On these series of studies, Sato et al. demonstrated that 1-SEAs created a funnel-shaped erosion beneath the adhesive–enamel interface17,18), thereby indicating a weak area that was vulnerable to acid attack. Moreover, they confirmed that these defects were not observed in the groups wherein primer was applied on PA–pre-etched enamel.

In this study, the influence of primer contamination on enamel bonding was analyzed. Adper Scotchbond Multi-Purpose (SMP), CLEARFIL SE Bond 2 (SE2), Scotchbond Universal (SBU) Adhesive, and Scotchbond Etchant (35% phosphoric acid; PA) were used. Ground bovine enamels were divided into eight groups based on the bonding protocols. The bonding interfaces after an acid–base challenge were observed via scanning electron microscopy to determine the acid–base resistant zone (ABRZ). Moreover, the bonding interfaces after the nanoleakage challenge were analyzed via energy-dispersive X-ray spectroscopy. ABRZ was observed in all the samples except PA_ONLY (applied PA etching, No primer and No adhesive resin). The funnel-shaped erosion was detected only in X_SBU (applied SBU without PA etching). The nanoleakage test revealed silver patterns in some groups. The nanoleakage, i.e., the penetration of the silver ions, was detected in the groups wherein primer was applied on PA–pre-etched enamel.

Keywords: Enamel, Phosphoric acid, Acid–base resistance zone, Nanoleakage
were kept under frozen storage conditions, were utilized. They were kept moist for 30 min at room temperature (23°C) to prevent dehydration before use. The labial surfaces were ground using a 280-grit SiC paper in running water until ~50-mm² area of the ground enamel was exposed. To standardize the surface roughness and smear layer, the specimens were ground using a 600-grit SiC paper in running water for 30 s. The materials employed in this study are listed in Table 1. Furthermore, Adper Scotchbond Multi-Purpose (SMP, 3M ESPE, St. Paul, MN, USA), CLEARFIL SE Bond 2 (SE2, Kuraray Noritake Dental, Tokyo, Japan), and Scotchbond Universal Adhesive (SBU, 3M ESPE) were also used. For the PA etching, the Scotchbond Etchant (35% PA, 3M ESPE) was applied for 15 s. The 96 specimens were randomly divided into eight groups (12 specimens per group) based on the bonding protocols (Table 2). Afterwards, the groups were divided into two subgroups (6 specimens per subgroup); one subgroup was provided to “Observation of ABRZ and the funnel-shaped erosion”, and another subgroup was provided to “nanoleakage test”. Each bonding procedure was performed, and the bonding surface was light-cured for 10 s. A light-emitting diode (LED) light-curing unit (VALO LED curing light, Ultradent Products, South Jordan, UT, USA) was employed in high-power mode (1,400 mW/cm²). Then, CLEARFIL MAJESTY LV (A2 shade, Kuraray Noritake Dental), which was ~2 mm thick, was placed between the two enamel specimens and light-cured for 40 s. **Observation of ABRZ and the funnel-shaped erosion**

The sample preparation is illustrated in Fig. 1. The bonded specimens were stored in deionized water for 24 h at 37°C. Next, each specimen was sectioned perpendicular to the bonding interface into two halves using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) and embedded in the epoxy resin (Epoxicure, Buehler). Then, they were polished using SiC paper (600–1200 grit), after which they were subjected to the acid–base challenge to examine ABRZ in the interfacial structure. For the acid challenge, each specimen was stored in buffered demineralized solution (pH 4.5, 2.2 mmol/L CaCl₂, 2.2 mmol/L NaH₂PO₄, and 50 mmol/L acetic acid) for 4.5 h. For the base challenge, the specimens were immersed in 6% NaOCl for 20 min with ultrasonic agitation to remove any debris from the demineralized structures of enamel proteins, followed by rinsing with running deionized water for 30 s.**

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**Table 1** Materials employed in this study

| Materials                      | Manufacturer             | Lot number | Main composition                                                                 |
|--------------------------------|--------------------------|------------|----------------------------------------------------------------------------------|
| Phosphoric acid                |                          |            |                                                                                  |
| Scotchbond etchant             | 3M ESPE, St. Paul, MN, USA | 7423       | 35% phosphoric acid                                                              |
| Bonding agent                  |                          |            |                                                                                  |
| Adper Scotchbond Multi-Purpose (SMP) | 3M ESPE                | Primer     | Primer: water, HEMA, polyalkenoic acid                                          |
|                                |                          | Adhesive resin | Adhesive: bis-GMA, HEMA, triphenylantimony, amines                                |
| CLEARFIL SE Bond 2 (SE2)       | Kuraray Noritake Dental, Tokyo, Japan | Primer | Primer: 10-MDP, HEMA, hydrophilic aliphatic dimethacrylate, di-camphorquinone, water |
|                                |                          | Adhesive resin | Adhesive: 10-MDP, Bis-GMA, HEMA, hydrophilic aliphatic dimethacrylate, di-camphorquinone, initiators, Accelerators, and silanated colloidal silica |
| Scotchbond Universal Adhesive (SBU) | 3M ESPE                | Primer     | Bis-GMA, 2-hydroxyethyl methacrylate decamethylene, dimethacrylate, ethanol, water, silane-treated silica, 2-propenoic acid 2-ethyl, reaction products with 1,10-decanediol and phosphorus, oxide, a copolymer of acrylic and itaconic acid, camphorquinone, and dimethylaminobenzoate |
|                                |                          | Primer     |                                                                                  |
| Resin composite                |                          |            |                                                                                  |
| CLEARFIL MAJESTY LV            | Kuraray Noritake Dental | Primer     | TEGDMA, hydrophobic aromatic dimethacrylate, camphorquinone, Silanated barium glass filler, and Silanated colloidal silica |
| A2 shade                       | 9D0027                   | Primer     |                                                                                  |
|                                |                          | Primer     |                                                                                  |

Bis-GMA: bisphenol A-glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-methacryloyloxydecyldihydrogen phosphate; TEGDMA: triethylene glycol dimethacrylate.
Table 2  Experiment groups and protocols

| PA etching          | Primer application                                             | Adhesive resin application |
|---------------------|---------------------------------------------------------------|----------------------------|
| PA_ONLY             | Scotchbond etchant was applied and left for 15 s, after which it was washed with water for 5 s. | None                       | None                       |
| PA_SMPpa            | Scotchbond etchant was applied and left for 15 s, after which it was washed with water for 5 s. | SMP primer was applied, followed by air blowing for 5 s. | SMP adhesive was applied, followed by air blowing for 5 s. |
| PA_SMPxa            | Scotchbond etchant was applied and left for 15 s, after which it was washed with water for 5 s. | None                       | SMP adhesive was applied, followed by air blowing for 5 s. |
| X_SE2               | None                                                          | SE2 primer was applied for 20 s, followed by air blowing for 5 s. | SE2 adhesive was applied, followed by air blowing for 5 s. |
| PA_SE2pa            | Scotchbond etchant was applied and left for 15 s, after which it was washed with water for 5 s. | SE2 primer was applied for 20 s, followed by air blowing for 5 s. | SE2 adhesive was applied, followed by air blowing for 5 s. |
| PA_SE2xa            | Scotchbond etchant was applied and left for 15 s, after which it was washed with water for 5 s. | None                       | SE2 adhesive was applied, followed by air blowing for 5 s. |
| X_SBU               | None                                                          | SBU was applied for 20 s, followed by air blowing for 5 s. |
| PA_SBU              | Scotchbond etchant was applied and left for 15 s, after which it was washed with water for 5 s. | SBU was applied for 20 s, followed by air blowing for 5 s. |

the surface to prevent wear or fracture of the remaining structure during polishing. Subsequently, the specimens were cut perpendicular to the adhesive–enamel interface and polished using SiC papers (600–2000 grit) and diamond pastes (Struers, Ballerup, Denmark) down to particle sizes of 0.25 μm. Moreover, argon-ion etching (EIS-200ER, Elionix, Tokyo, Japan) was performed on the polished surfaces to sharply relieve the adhesive–enamel interface with an accelerating voltage of 1 kV and an ion current density of 1.5 mA/cm² for 30 s. The specimens were finally gold sputter-coated, and the morphological structure of the interface was observed after the acid–base challenge via scanning electron microscopy (SEM, JSM-IT100, JEOL, Tokyo, Japan) at accelerating voltage and magnification of 20 kV and 1,500×, respectively.

**Fig. 1**  Sample preparation for the observations of ABRZ and funnel-shaped erosion.

**Nanoleakage test**

The nanoleakage patterns at the adhesive–enamel interface were evaluated for each experimental group (Table 2). The sample preparations for the nanoleakage test are illustrated in Fig. 2. The bonded specimens were
Fig. 2 Sample preparation for the nanoleakage test.

stored in deionized water for 24 h at 37°C. Then, the samples were embedded in the epoxy resin (EpoxiCure). Next, the epoxy resin was cured, and the specimens were vertically sectioned at the center and divided into two halves using an Isomet sectioning saw. Then, the specimens were immersed in 50% ammoniacal silver nitrate (AgNO₃) solution for 24 h in darkness. The specimens were thoroughly rinsed with deionized water for 5 min and placed in a photo-developing solution for 8 h under fluorescent light to reduce the penetration of the metallic silver grains by the silver ions. Subsequently, the specimens were polished using 2000-grit SiC paper and diamond pastes down to a particle size of 0.25 μm. Then, the specimens were gold sputter-coated. Finally, the morphological structure of the bonding interface was observed via SEM and energy-dispersive X-ray spectroscopy (EDS; JSM-IT100) at a working distance of 10 mm and an accelerating voltage of 20.0 kV, employing a 2,000× magnification.

RESULTS

Observation for ABRZ and the funnel-shaped erosion
The interfacial morphologies of each group after the acid–base challenge are shown in Fig. 3. Outer lesions (OLs) created by the mineral loss due to the acid–base challenge were observed in all the groups. The OL depth ranged from 25 to 30 μm in all the groups. After the acid–base challenge, ABRZ, a residual enamel-like structure that was detected beneath the bonding interface, was observed in all the samples except PA_ONLY (Fig. 3a). The thickness of ABRZ in each group was ~6, 4, 2, 6, 4, 1, 3 μm (Figs. 3b–h). ABRZs in PA_SE2pa and PA_SE2xa (Figs. 3e and f) were thicker than that of X_SE2 (Fig. 3d). Similarly, ABRZ was thicker in PA_SBU (Fig. 3h) compared to X_SBU (Fig. 3g). Meanwhile, the funnel-shaped erosion beneath the interface was observed only in X_SBU (Fig. 3g).

Nanoleakage test
The representative SEM and EDS images of the
ammoniacal-immersed AgNO₃-challenged specimens of the adhesive–enamel interface are illustrated in Figs. 4 and 5, respectively. The silver patterns, i.e., nanoleakages, at the bonding interfaces of PA_SMPpa, PA_SE2pa, and PA_SBU (Figs. 4b, e, and h, respectively), were observed via SEM. Moreover, EDS analysis detected the silver patterns in PA_SMPpa, PA_SE2pa, X_SBU, and PA_SBU (Figs. 5b, e, g, and h, respectively). For PA_SMPpa, PA_SE2pa, and PA_SBU, distinctive silver patterns were observed at the adhesive–enamel interface to a depth of ~5 μm, following the enamel prism orientation (Figs. 5b, e, and h). On the contrary, the silver pattern of X_SBU was observed only at the adhesive–enamel interface (Fig. 5g, arrow).

DISCUSSION

Previous studies have utilized SEA to reveal ABRZ beneath the enamel bonding interface.⁶⁻²⁻³. In those studies, the residual enamel-like structures were detected beneath the bonding interface after the acid-base challenge, although an intact enamel substrate was dissolved by the challenge (OL was created). Therefore, ABRZ was believed to be crucial in inhibiting the damage from recurrent caries, by sealing the restoration margins, thus promoting the durability of restoration by maintaining the bond.⁶⁻²⁻³. Here, the enamel ABRZ was detected in all the groups, except in PA_ONLY. These observations indicate that the micromechanical interlocking between the etched enamel and base monomer of the resin composite, i.e., without the application of the adhesive resin, is a shortcoming that prevents secondary caries. Therefore, the penetration of enamel (tooth) by monomer substrates might be crucial in establishing a stable bonding interface. Moreover, the funnel-shaped erosion was also observed next to ABRZ, only in X_SBU (Fig. 3g). Additionally, even if ABRZ in the enamel was detected, this erosion still indicated the
existence of an area susceptible to an acid attack at the bonding interface. In contrast, this defect was improved by PA etching prior to the application of SBU (Fig. 3h). These results are consistent with those of previous studies, which employed several 1-SEAs for enamel bonding\cite{17,18}. Moreover, a silver pattern, indicating a nanoleakage, was also detected via EDS in PA_SBU and X_SBU (Figs. 5h and g, respectively). This new finding implied that the defects of the 1-SEA bond with enamel were not completely overcome by PA etching.

Several in vitro studies have indicated that the etching efficacy of SEA systems may be too weak to adequately etch the smear layer and enamel\cite{28-31}. Furthermore, those studies reported that SEA with a pre-PA etching group exhibited a significantly higher bond strength than the group without pre-PA etching\cite{32-34}. Thus, some clinicians have performed selective enamel etching to improve the enamel bonding of mild, and especially ultra-mild, SEAs\cite{5,35}. It was assumed that the bond strength of SEA systems that are employed in pre-etching enamel with PA would be equivalent to that obtained with the Etch&Rinse systems bonded to the enamel\cite{36}. In this study, however, X_SE2 did not exhibit funnel-shaped erosion and nanoleakage image. In other words, SE2 created a stable bonding interface without PA etching. In contrast, a nanoleakage was detected in PA_SE2pa. Moreover, PA_SE2xa and X_SE2 also facilitated the stable bonding interface. When considering the difference between PA_SE2pa and PA_SE2xa, the application of a primer might affect the bonding to enamel that had been pre-etched with PA. Rathke et al.\cite{37} demonstrated that unavoidable contamination of pre-etched enamel by a primer could affect the enamel bond as the bond strength of the pre-etched enamel with 2-SEA would have decreased in the priming step compared to the procedure without the priming step.

To demonstrate the influence of the primer, SMP was utilized. SMPs are one of the adhesives that are presently referred to as the three-step Etch&Rinse system because they involve the application of a conditioner, a primer, and an adhesive resin in three application steps. With the depositions of silver, a nanoleakage was observed in PA_SMPpa (Fig. 4b, arrow). In contrast, the leakage was not detected in PA_SMPxa (Fig. 4c). For PA_SMPxa, the primer was not applied to the surface of the enamel. This procedure is based on the manufacturer’s instructions for enamel treatment. Additionally, ABRZs without the funnel-shaped erosion were observed in both PA_SMPpa and PA_SMPxa (Figs. 3b and c). However, no nanoleakage was detected in PA_ONLY in which both the primer and adhesive resin were not applied.

Comparing the three cases, the application of the primer to a pre-etched enamel might result in nanoleakage at the adhesive–enamel interface. Water and 2-hydroxyethyl methacrylate (HEMA)-rich solutions that ensure the complete expansion of the dentin collagen fibril meshwork and wet the collagen with hydrophilic monomers are the generally employed primers\cite{38}, which were also adopted in this study. It was reported that air-drying could be inadequate to remove water from the micropores of etched enamel because evaporation increases the proportion of HEMA in HEMA/water mixtures, thus reducing the vapor pressure of water\cite{39}. It is assumed that deep jaggedness created by PA etching on the enamel surface renders water and solvent evaporations a challenge. Therefore, these residual hydrophilic solvents might compete with the hydrophobic monomers that are contained in adhesive resin, e.g., Bis-GMA\cite{37,39}, which triggers a nanoleakage. Therefore, the EDS results, which demonstrated that the silver ions had penetrated the enamel, indicate the existence of a residual primer at the micropores of etched enamel in PA_SMPpa, PA_SE2pa, and PA_SBU (Figs. 5b, e, and h, respectively). Different from these three groups, the silver pattern of X_SBU was observed only at the adhesive–enamel interface. Generally, SBU is classified as an ultra-mild 1-SEA, which indicates the effect of low etching on enamel\cite{18,49}. Therefore, this inferior enamel bonding might result in a nanoleakage between the enamel and adhesive resin.

The results of this study require a rejection of the null hypothesis, that the application of the primer did not influence the enamel bonding. Here, the stable bonding interface, ABRZ without erosion and nanoleakage, was observed in PA_SMPxa, X_SE2, and PA_SE2xa. In the case of PA_SMPxa and PA_SE2xa, primer application was unnecessary for enamel surface. However, primer application was necessary for dentin surface. Generally, the tooth cavity for the treatment of caries possesses both the enamel and dentin surfaces. In the clinical setting, the strict selective applications for enamel and dentin require high-technique sensitivity. To this end, X_SE2 could treat both enamel and dentin using the same method, namely by total etching. Generally, it is presently agreed that the most favorable bonding performance to dentin is obtained with mild SEAs\cite{9}. Many clinical trials have reported high success rates with mild 2-SEA\cite{41-45}. Therefore, the application of SE2 and mild 2-SEA without pre-etching may be acceptable in clinical procedures although several in vitro studies have reported that pre-PA etching had statistically improved the enamel bond strength\cite{32-34}. This study was performed on ground enamel surface. Meanwhile, bonding procedure is sometimes applied to uncut enamel in the clinical situation. Hence, further studies are needed to clarify the bonding performance on uncut enamel.

**CONCLUSION**

Within the limits of the present study, the primer application on enamel did not influence the formation of enamel ABRZ. Conversely, nanoleakage, which is the penetration of silver ions, was detected in the groups wherein primer was applied on PA–pre-etched enamel.

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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) Bertolotti RL. Total etch: The rational dentin bonding protocol. J Esthet Dent 1991; 3: 1-6.
3) Fusayama T, Nakamura M, Kurosaki N, Iwaku M. Non-pressure adhesion of a new adhesive restorative resin. J Dent Res 1979; 58: 1364-1370.
4) Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: Current status and future challenges. Oper Dent 2003; 28: 215-235.
5) Van Meerbeek B, Yoshihara K, Van Landuyt K, Yoshida Y, Peumans M. From Buonocore’s pioneering acid-etch technique to self-adhering restoratives: A status perspective of rapidly advancing dental adhesive technology. J Adhes Dent 2020; 22: 7-34.
6) Akimoto N, Takamizawa M, Momoi Y. 10-year clinical evaluation of a self-etching adhesive system. Oper Dent 2007; 32: 3-10.
7) Peumans M, De Munck J, Van Landuyt K, Van Meerbeek B. Thirteen-year randomized controlled clinical trial of a two-step self-etch adhesive in non-caries lesions. Dent Mater 2015; 31: 308-314.
8) de Goes MF, Shinohara MS, Freitas MS. Performance of a new one-step multi-mode adhesive on etched vs non-etched enamel on bond strength and interfacial morphology. J Adhes Dent 2014; 16: 243-250.
9) Marchesi G, Frassetto A, Mazzoni A, Apolonio F, Diolosà M, Cadenaro M, et al. Adhesive performance of a multimode adhesive system: 1-year in vitro study. J Dent 2014; 42: 603-612.
10) Frankenberger R, Krämer N, Petschelt A. Long-term effect of dentin primers on enamel bond strength and marginal adaptation. Oper Dent 2000; 25: 11-19.
11) Moll K, Haller B, Hofmann N. Initial composite-enamel bond strength of current adhesive techniques. Acta Med Dent Helv 1997; 2: 233-240.
12) Hadavi F, Hey JH, Ambrose ER, Louie PW, Shinkewski DJ. The effect of dentin primer on the shear bond strength between composite resin and enamel. Oper Dent 1993; 18: 61-65.
13) Hotta M, Kondoh K, Kamemizu H. Effect of primers on bonding agent polymerization. J Oral Rehabil 1998; 25: 792-799.
14) Jain P, Stewart GP. Effect of dentin primer on shear bond strength of composite resin to moist and dry enamel. Oper Dent 2000; 25: 51-58.
15) Thoms LM, Nichols JI, Brudvik JS, Kydd WL. The effect of dentin primer on the tensile bond strength to human enamel. Int J Prosthodont 1994; 7: 403-409.
16) Nikaido T, Takagaki T, Sato T, Burrow MF, Tagami J. The concept of super enamel formation —Relationship between chemical interaction and enamel acid-base resistant zone at the self-etch adhesive/enamel interface. Dent Mater J 2020; 39: 534-538.
17) Sato T, Takagaki T, Ikeda M, Nikaido T, Burrow MF, Tagami J. Effects of selective phosphoric acid etching on enamel using “no-wait” self-etching adhesives. J Adhes Dent 2018; 20: 407-415.
18) Sato T, Takagaki T, Matsui N, Hamba H, Sadr A, Nikaido T, et al. Morphological evaluation of the adhesive/enamel interfaces of two-step self-etching adhesives and multimode one-bottle self-etching adhesives. J Adhes Dent 2016; 18: 223-229.
19) Halabi S, Matsui N, Nikaido T, Abdo A, Burrow MF, Tagami J. Effect of two bleaching regimens on enamel bonding performance. Dent Mater J 2020; 2019-230.
20) Halabi S, Matsui N, Nikaido T, Burrow MF, Tagami J. Effect of office bleaching on enamel bonding performance. J Adhes Dent 2019; 21: 167-177.
21) Kakiuchi Y, Takagaki T, Ikeda M, Sato T, Matsui N, Nikaido T, et al. Evaluation of MDP and NaF in two-step self-etch adhesives on enamel microshear bond strength and morphology of the adhesive-enamel interface. J Adhes Dent 2018; 20: 527-534.
22) Li N, Nikaido T, Alireza S, Takagaki T, Chen JH, Tagami J. Phosphoric acid-etching promotes bond strength and formation of acid-base resistant zone on enamel. Oper Dent 2013; 38: 82-90.
23) Li N, Nikaido T, Takagaki T, Sadr A, Makishi P, Chen J, et al. The role of functional monomers in bonding to enamel: Acid-base resistant zone and bonding performance. J Dent 2010; 38: 722-730.
24) Tay FR, Fashley DH, Yoshiyama M. Two modes of nanoleakage expression in single-step adhesives. J Res 2002; 81: 472-476.
25) Nikaido T, Ichikawa C, Li N, Takagaki T, Sadr A, Yoshida Y, et al. Effect of functional monomers in all-in-one adhesive systems on formation of enamel/dentin acid-base resistant zone. Dent Mater 2011; 30: 576-582.
26) Nikaido T, Narrohman H, Takagaki T, Sadr A, Ichinose S, Tagami J. Nanoleakage in hybrid layer and acid-base resistant zone at the adhesive/dentin interface. Microsc Microanal 2015; 21: 1271-1277.
27) Nikaido T, Weerasinghe DD, Waidyasekera K, Inoue G, Foxton RM, Tagami J. Assessment of the nanostructure of acid-base resistant zone by the application of all-in-one adhesive systems: Super dentin formation. Biomed Mater Eng 2009; 19: 163-171.
28) Abdalla AI, El Zohairy AA, Mohsen MA, Feilzer AJ. Bonding efficacy and interface morphology of self-etching adhesives to ground enamel. J Adhes Dent 2010; 12; 19-25.
29) Osorio R, Monticelli F, Moreira MAG, Osorio E, Toledano M. Enamel resin bond durability of self-etch and etch & rinse adhesives. Am J Dent 2009; 22: 371-375.
30) Torres CR, Barcellos DC, Pucci CR, Lima Gde M, Rodrigues CM, Siviero M. Influence of methods of application of self-etching adhesive systems on adhesive bond strength to enamel. J Adhes Dent 2009; 11: 279-286.
31) Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. Dent Mater 2011; 27: 17-28.
32) Erickson RL, Barkmeier WW, Latta MA. The role of etching in bonding to enamel: A comparison of self-etching and etch-and-rinse adhesive systems. Dent Mater J 2009; 25: 1459-1467.
33) Frankenberger R, Lohbauer U, Roggendorf MJ, Naumann M, Taschner M. Selective enamel etching reconsidered: Better than etch-and-rinse and self-etch? J Adhes Dent 2008; 10: 339-344.
34) Takamizawa T, Barkmeier WW, Tsujimoto A, Endo H, Tsuchiya K, Erickson RL, et al. Influence of pre-etching times on fatigue strength of self-etching adhesives to enamel. J Adhes Dent 2016; 18: 501-511.
35) Rosa WL, Fiva E, Silva AF. Bond strength of universal adhesives: A systematic review and meta-analysis. J Dent 2015; 43: 765-776.
36) Erickson RL, Barkmeier WW, Kimmes NS. Bond strength of self-etch adhesives to pre-etched enamel. Dent Mater 2009; 25: 1187-1194.
37) Rathke A, Ostermeyer Y, Muche R, Haller B. Reconsidering the double etching of enamel: do self-etching primers contaminate phosphoric acid-etch enamel? J Adhes Dent 2013; 15: 107-114.
38) Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M, et al. State of the art etch-and-rinse adhesives. Dent Mater 2011; 27: 1-16.
39) Moll K, Gärtner T, Haller B. Effect of moist bonding on composite/enamel bond strength. Am J Dent 2002; 15: 85-90.
40) Wong J, Tsujimoto A, Fischer NG, Baruth AG, Barkmeier WW, Johnson EA, et al. Enamel etching for universal adhesives: Examination of enamel etching protocols for optimization of bonding effectiveness. Oper Dent 2020; 45: 80-91.
41) Abdalla AI, García-Godoy F. Clinical evaluation of self-etch adhesives in Class V non-curious lesions. Am J Dent 2006; 19: 289-292.
42) Burrow MF, Tyas MJ. Clinical evaluation of three adhesive systems for the restoration of non-curious cervical lesions. Oper Dent 2007; 32: 11-15.
43) Perdigão J, Carmo AR, Anauate-Netto C, Amore R, Lewgoy HR, Cordeiro HJ, et al. Clinical performance of a self-etching adhesive at 18 months. Am J Dent 2005; 18: 135-140.
44) Türkün SL. Clinical evaluation of a self-etching and a one-bottle adhesive system at two years. J Dent 2003; 31: 527-534.
45) Yaman BC, Doğruer I, Gümüştaş B, Efes BG. Three-year randomized clinical evaluation of a low-shrinkage silorane-based resin composite in non-curious cervical lesions. Clin Oral Investig 2014; 18: 1071-1079.