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Original Article

Effects of Rotary Brush Active Application on Enamel Etching Patterns of Self-Etch Adhesive Systems

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Abstract

Aim: The aim of the present study was to assess the effects of rotary brush active application technique on the ultramorphological changes induced by self-etching adhesive systems on ground enamel.

Materials and methods: Three application methods were deployed: (1) passive application; (2) manual active application; (3) rotary-brush active application. Two self-etching adhesive systems were used: (1) Optibond All-in-one; (2) Nova Combo Plus. Acid-etching of the ground enamel surface with phosphoric acid was used as control. Labial surfaces of bovine incisors were separated into four pieces. One of three application methods including phosphoric acid-etching was used to apply one of self-etch adhesive systems to enamel surface of each enamel piece respectively. Treated enamel samples were then processed for evaluation under scanning electron microscope (SEM).

Results: SEM findings showed that acid-etching completely removed the smear layer from the ground enamel surface. None of the active application techniques, however, were able to eliminate the smear layers.

Conclusions: Active application of self-etch adhesives to enamel with a rotary brush may not have an effect on the interaction of the tested self-etch adhesives with smear layers on the enamel surface.

Keywords

dental adhesives, enamel, morphological, SEM, self-etch

INTRODUCTION

Traditionally, the conditioning of enamel prior to resin bonding has been carried out by phosphoric acid, a conventional technique called "acid-etch technique" used since 1955.[1] This clinical technique enables micromechanical locking between the enamel surface and the resin material by removing the smear layers that occur during the cavity preparation on the enamel surface and creating superficial demineralization on the enamel surface. As a result of this interaction between the enamel surface and phosphoric acid, the expansion occurs in the interprismatic spaces that enable the resin tag formation. It is assumed that mechanical retention of the resin-enamel bonding is achieved by the formation of resin tags.[1-4] With the introduction of acid-etch technique, adhesive dentistry has changed with traditional cavity preparation techniques with more conservative cavity preparation techniques.[2]

Self-etch adhesives, which involve fewer clinical processes and are hence more user-friendly, have been developed as an alternative to total-etch adhesives that employ the acid-etching approach.[5] The characteristic feature of self-etch adhesives is that their chemical content contains polymerisable acidic monomers that both condition and prime enamel and dentine at the same time.[6] The most recent form of these materials combines etchant, primer, and adhesive into one bottle, that etches tooth surfaces and polymerises at the same time. Benefits like shorter application time and lower technique sensitivity are rumoured for self-etch adhesive systems.[6,7]

Although initial dentin bonding performances of current self-etch adhesive systems are generally acceptable, the success of enamel bonding is still controversial.[8] Since self-etch adhesives are less acidic than phosphoric acid, their demineralisation abilities, and therefore, their ability to create microporosity on the enamel surface are more limited. Although no significant difference was found in some studies between the enamel bond strength of total-etch adhesives and self-etch adhesive systems[9,10], most studies showed that phosphoric acid pre-etching significantly increased the enamel bond strengths of self-etch adhesives.[8,11,12]

Different methods have been proposed to overcome the problem of reduced bonding capacity of self-etch adhesives to enamel. The foremost commonly used strategy is that of pre-etching of enamel with phosphoric acid.[12] Also, whereas self-etch adhesive was applied to the enamel with micro-brush agitation, in alternative words, active application method was reported to enhance enamel bonding for some adhesives.[13,14] Additionally to those ways, it has been reported that active application of self-etch adhesives with sonic instruments to enamel offers positive results.[15,16] Recently, it has been reported that active application of self-etch adhesive to enamel with the aid of rotary brush increases the bond strengths of two different self-etch adhesive systems to enamel.[17] The authors suggested that the application of self-etch adhesive to the enamel with a rotary brush might increase the ability of the self-etch adhesive to dissolve the smear layer, thus improving their enamel etching capacities. However, there is no morphological proof to support this claim.

AIM

The objective of the present study was to evaluate morphological aspects of enamel surfaces which are treated with different self-etch adhesives by either one of these application techniques including, passive application, manual active application and rotary brush active application. The hypothesis to be tested was that the etching patterns produced on ground enamel is not similar when phosphoric acid and different application methods of self-etching systems are compared.

MATERIALS AND METHODS

Two commercial single-step self-etch adhesive systems, Optibond All-in-one, and Nova Combo Plus, and three application methods, passive application, manual active application, and rotary brush active application were used as shown in **Table 1**.

The crowns of eight bovine teeth were separated into four pieces to obtain enamel rectangles. Ground enamel surfaces were prepared by polishing all enamel samples under water with 600-, 800-, 1000-, 1500-, 2000-grit silicon carbide (SiC) abrasive papers for 30 s. Enamel rectangles were divided into eight groups (n=4) according to the adhesive brand (Optibond All-in-one; Nova Combo Plus) and the application technique (passive application, manual active application, and rotary-brush active application).

For the SEM analysis, the self-etch adhesive systems were applied to enamel surfaces according to manufacturer instructions under the various test conditions **(Table 1)**; however, they weren't photo-polymerised. The enamel surfaces were then directly stirred in acetone for twenty-four

Table 1. Materials and application methods used in the study

Adhesive system	Chemical composition	pH
Optibond All-in-one	GPDM, HEMA, GDMA, Bis-GMA, water, acetone, ethanol, CQ, silica filler	2.5 - 3.0
Nova Combo Plus	Bis-GMA, HEMA, ethanol, 10-MDP, 4-META, silanated nano silica, initiators, water	2.5 - 2.7

Application methods	
Passive application	Left undisturbed one coat of adhesive for 20 s. Gently air thin for 5 s.
Manual active application	Agitate one coat of adhesive for 20 s. Gently air thin for 5 s.
Rotary-brush active application	Agitate one coat of adhesive for 20 s by using low-speed rotary brush at the speed of 1000 rpm.

Abbreviations: 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; 4-META: 4-methacryloxyethyl trimellitate anhydride, GPDM: glycerol phosphate dimethacrylate.

hours to dissolve the resin materials from the enamel surface in dark bottles.[18] A group of etching enamel surfaces with 37% orthophosphoric acid for 15 s served as a control. All enamel samples were dried under vacuum for twenty-four hours, then mounted on aluminum stubs with carbon tape, sputter-coated with gold-palladium and evaluated in scanning electron microscope (SEM, Zeiss EVLO LS10, Bruker, Bremen, Germany) at an accelerating voltage of 10.0 kV and a working distance of 8.5–10.5 mm.

RESULTS

In the control group, etching the ground enamel surface with phosphoric acid for 20 seconds resulted in the appearance of enamel prisms without any sign of smear layers **(Fig. 1a)**. Polishing scratches were observed along the enamel surface in all groups where the Optibond All-in-one adhesive system was applied **(Fig. 1)**. It was observed that the application of the Optibond All-in-one adhesive system with rotating brush or manual active application techniques did not cause any change in the structure of the smear layer on the enamel surface. Enamel surfaces were largely unetched in all groups. As with the Optibond All-in-one adhesive system, different enamel surfaces were seen in all groups where Nova Combo Plus was applied with the control group. Polishing scratches remained intact in all Nova Combo Plus groups. This showed that the smear layer changed with Nova Combo Plus application in any group **(Fig. 2)**.

DISCUSSION

Phosphoric acid application, i.e. etching, on enamel surfaces covered with smear layer dissolves and removes the smear layer from the enamel surface and creates microporosities on the enamel surface by demineralization. When the low viscosity adhesive resin flows into these microporosities and polymerises in these areas, it provides a mechanical retention with the enamel tissue.[4] In this electron microscope study, it was observed that after etching the enamel surfaces covered with the smear layer with phosphoric acid, the smear layer disappeared from the surface, microporosities formed on the enamel surface, and enamel prisms were revealed.

It has often been reported that self-etch adhesives, which do not have the step of etching enamel with phosphoric acid in clinical practice procedures, do not act like phosphoric acid on the enamel surface. It is known that mild self-etch adhesives with generally used pH values between 2 and 2.7 have low effectiveness in removing the smear layers on the enamel surface and therefore their enamel bonding strength is low.[19,20] This difference in etching patterns was also observed in the SEM evaluation in this study. It was observed that the mild self-etch adhesives Optibond

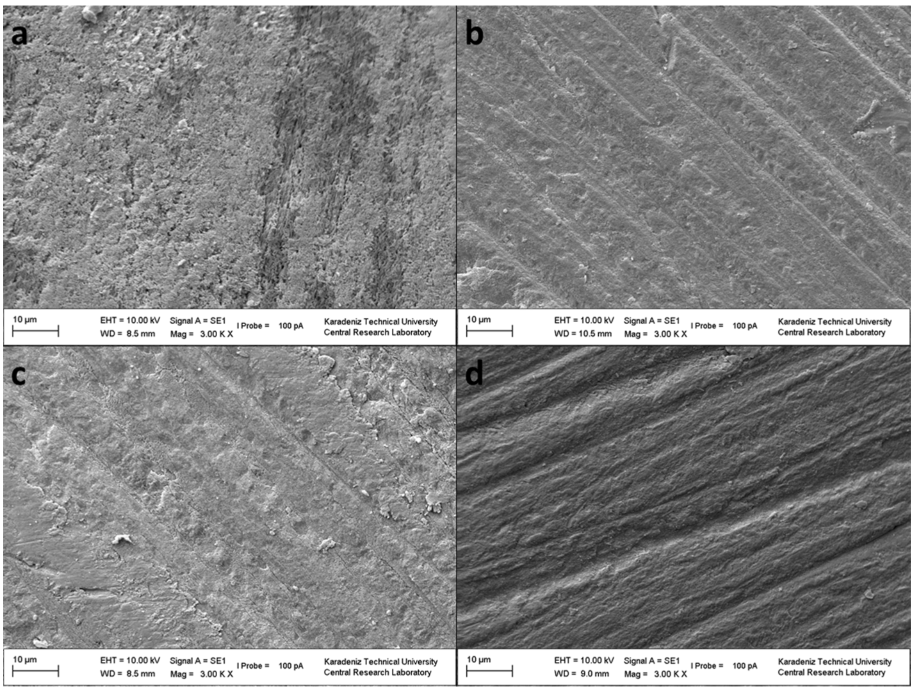

Figure 1. Representative scanning electron microscopy images of permanent enamel after treatment with 37% orthophosphoric acid for 20 s (**a**); appearances of permanent enamel after treatment with Optibond All-in-one; passive application (**b**), manual active application (**c**), and rotary-brush active application (**d**). Phosphoric acid etched enamel showed no sign of smear layer and more irregular and rough surface properties (**a**) in comparison to enamel treated with Optibond All-in-one self-etch adhesive alone (**b**) or with both active application techniques (**c**, **d**).

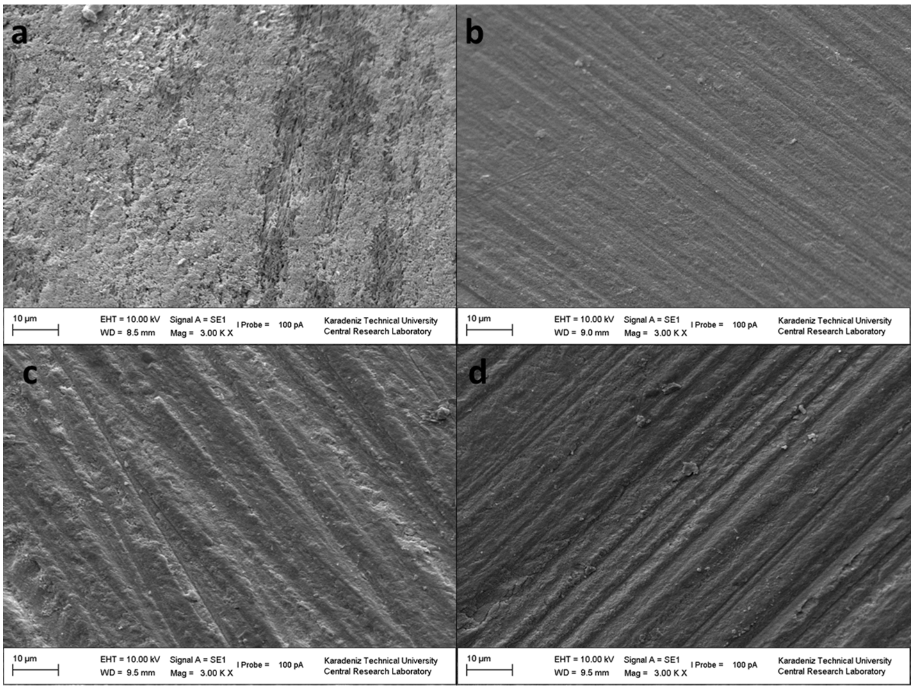

Figure 2. Representative scanning electron microscopy images of permanent enamel after treatment with 37% orthophosphoric acid for 20 s (**a**); appearances of permanent enamel after treatment with Nova Combo Plus; passive application (**b**), manual active application (**c**), and rotary-brush active application (**d**). Phosphoric acid etched enamel showed no sign of smear layer and more irregular and rough surface properties (**a**) in comparison to enamel treated with Nova Combo Plus self-etch adhesive alone (**b**) or with both active application techniques (**c**, **d**).

All-in-one (pH: 2.5-3.0) and Nova Combo Plus (pH: 2.5-2.7) did not effectively condition the smear layer covering the enamel surfaces, thus creating a less retentive enamel surface morphology for enamel bonding.

One of the reasons why enamel bonding performances of self-etch adhesives are generally not as good as dentin bonding is that self-etch adhesives cannot produce sufficient demineralisation on the enamel surface covered with the smear layer. Therefore, many researchers have reported that etching the enamel surface with phosphoric acid before applying self-etch adhesives to enamel can increase bond strength to enamel.[11,12] In addition, it has been reported that active application of self-etch adhesives to enamel, in other words, agitation adhesive with microbrush manually during application increases enamel bond strength.[14] In previous studies, it was suggested that active application can increase the bonding performance of self-etch adhesives to enamel by increasing the demineralisation ability of the self-etch adhesive and/or reducing the amount of residual solvent.[14] However, in SEM analyses, it has been reported that the active application technique does not cause any change in the etching pattern that adhesives create on the enamel surface.[14] Similarly, it was found that active application of self-etch adhesives tested in this study had no effect on the etching patters of adhesives.

Some researchers have used a sonic device[21], or an electrical device[22], or a low-speed rotary brush[17] to enhance the benefits provided by the active application method. In one study, self-etch adhesives were applied to enamel surfaces by active application with a microbrush attached to a sonic device at 170 Hz frequency. According to the findings of this study, the sonic application technique changed the etching patterns of self-etch adhesives on the enamel surface and enabled the adhesives to dissolve better the smear layer.[21] The authors suggested that sonic vibration applied to the microbrush could create vibrations and microscopic bubbles within the adhesive solution, allowing more fresh acidic monomers to come into contact with the enamel surface, thereby helping the deeper demineralization.

In a previous study, it was reported that active application of self-etch adhesive to enamel with a prophylaxis brush attached to a low-speed rotary angle which is found in every clinic increased the enamel bond strengths of the tested adhesives.[17] In the present study, the effect of this new technique on the morphology created by self-etch adhesives on the smear-coated enamel surface was investigated. The findings revealed that this new technique is not different from the manual active application technique in terms of the effects of self-etch adhesives on the enamel surface. Therefore, the reason this technique increases the enamel bond strength of self-etch adhesives may be that agitating the adhesive with a rotary brush significantly removes the residual solvent amount.

Obviously, it can be thought that agitating the adhesive solution with a rotary brush can provide the recirculation of fresh acidic monomers in the solution, allowing the adhesive to bring deeper demineralization. However, the speed of the rotating brush may cause the solvent to evaporate before this process occurs. In this study, the rotating brush was operated at a speed of 1000 rpm. Therefore, the effect of using rotating brushes at lower speeds on enamel bonding and etching patterns of self-etching adhesives should be investigated in further studies.

CONCLUSIONS

According to the findings of the present study, the following conclusions can be reached:

- Etching the enamel surfaces covered with smear layers with phosphoric acid for 20 seconds causes the smear layer to move away from the enamel surface and reveal the enamel prism and crystals.
- The application of mild self-etch adhesives to the enamel surfaces covered with smear layers does not remove the polishing scratches indicating the presence of smear layer on the enamel surface and does not cause the exposure of enamel prisms.
- Active application of self-etch adhesives manually or with a rotary brush did not cause deeper demineralisation on the enamel surface with the parameters in the present study.

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Влияние активного применения вращающейся щётки на рисунки травления эмали самопротравливающих адгезивных систем

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Резюме

Цель: Целью настоящего исследования было оценить влияние техники активного применения вращающейся щётки на ультраморфологические изменения, вызванные самопротравливающими адгезивными системами на отшлифованной эмали.

Материалы и метод: Были применены три метода нанесения: (1) пассивное применение; (2) ручное активное приложение; (3) активное применение вращающейся щётки. Были использованы две самопротравливающие адгезивные системы: (1) Optibond All-in-one; (2) Nova Combo Plus. В качестве контроля использовали кислотное протравливание отшлифованной поверхности эмали фосфорной кислотой. Губные поверхности бычьих резцов были разделены на четыре части. Для нанесения одной из самопротравливающих адгезивных систем на поверхность эмали каждого элемента эмали использовали один из трёх методов нанесения, включая протравливание фосфорной кислотой. Образцы обработанной эмали затем обрабатывали для оценки под сканирующим электронным микроскопом (SEM).

Результаты: Результаты SEM показали, что кислотное протравливание полностью удалило смазанный слой с поверхности эмали. Однако ни один из активных методов нанесения не смог устранить смазанные слои.

Заключение: Активное нанесение самопротравливающих адгезивов на эмаль вращающейся щёткой может не влиять на взаимодействие испытуемых самопротравливающих адгезивов со смазанными слоями на поверхности эмали.

Ключевые слова

стоматологические адгезивы, эмаль, морфологический, SEM, самопротравливающий