Effect of Active Pretreatment of Self-etching Primers on the Ultramorphology of Intact Primary and Permanent Tooth Enamel

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ABSTRACT

Purpose: The objective of this study was to evaluate the ultramorphological changes after agitated and nonagitated application of self-etching primer systems on unground primary and permanent enamel.
Methods: Five self-etching primer systems were used: (1) Clearfil SE Bond; (2) Clearfil Protect Bond; (3) Adper Prompt; (4) Xeno III; and (5) nonrinse conditioner (NRC). Noncarious human primary and permanent incisors were collected and stored frozen until used. Intact labial surfaces were divided into 2 halves, applying the self-etching primers with (active application) and without (inactive agitation) using a microbrush within the times recommended by the manufacturers. Treated surfaces were further processed for ultramorphological evaluation under scanning electron microscope (SEM).
Results: All tested self-etching systems produced weaker etch patterns and less dissolution of enamel surface compared with acid-etched samples in both primary and permanent teeth. Except for NRC and Adper Prompt, agitation of the surface did not increase the etching efficacy. Clearfil SE Bond and Clearfil Protect Bond resulted in similar morphological features following application in either mode.
Conclusion: While agitation of self-etching primers may improve etching efficacy, this effect appears to be dependent on the material used. Phosphoric acid produces well-defined etching patterns on intact primary and permanent enamel. (J Dent Child 2006;73:86-90)
KEYWORDS: SELF-ETCING PRIMER, ACID-ETCHING DENTAL, DENTAL ENAMEL, PRIMARY TOOTH, SCANNING ELECTRON MICROSCOPY

Significant improvements in enamel and dentin adhesive resins have made resin composites progressively more the material of choice for the restoration of both permanent and primary teeth. Unlike nonadhesive restorations that require specific cavity configurations, retention of adhesive restorations is achieved micromechanically by virtue of specific surface treatment methods. Since 1955,[1] the acid etch technique has been used with success to achieve micromechanical retention and, thus, adequate bond strength to tooth structures. Traditionally, enamel surface has been prepared by etching with phosphoric acid at concentrations ranging from 30% to 50% for 15 to 60 seconds, followed by rinsing and drying of the surface.[2-4]

Lately, with the demands of simplified application and reduction of chair-side time, self-etching adhesive systems have been introduced to the market.[5-7] On the scientific side, these materials were initially developed to overcome some of the shortcomings of total-etch systems, such as poor impregnation of the demineralized dentin layer. Through use of chemically modified acidic monomers, self-etch systems demineralize and penetrate dental hard tissues simultaneously without the requirement of a separate etching/rinsing/drying step. Although self-etch systems have clinically been claimed to reduce technique sensitivity, questions have surfaced regarding the reliability of these systems to achieve durable bonds to enamel comparable to those achieved traditionally with phosphoric acid etching.[8,9] Long-term

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resistance to microleakage in enamel is another problem requiring evaluation.

For a long time, the morphology and composition of primary and permanent teeth were considered to be similar and the knowledge about permanent teeth was transferred to primary teeth.10 Results of recently published investigations, however, permit the conclusion that, besides the obvious differences in terms of number, appearance, size, and shape, significant differences in the chemical and morphologic structures of the 2 dentitions do exist.[11,12]

The objective of this study was to examine the ultramorphological characteristics of unground (intact) primary and permanent tooth enamel after application of various self-etching systems by either: (1) agitating the enamel surface (active application); or (2) leaving it undisturbed.

METHODS

The self-etching systems selected for this study were: (1) Clearfil Protect Bond (Kuraray, Osaka, Japan); (2) Clearfil SE Bond (Kuraray, Osaka, Japan); (3) Adper Prompt (3M-ESPE, Seefeld, Germany); and (4) Xeno III (Dentsply, Konstanz, Germany). A nonrinse conditioner (NRC; Dentsply, Konstanz, Germany) and a 35% phosphoric acid gel (Scotchbond Etchant, 3M, St. Paul, Minn) were also used for comparisons. The compositions of the self-etch systems are shown in Table 1. Exfoliated, caries-free human primary central incisors (n=20) and permanent incisors extracted for periodontal reasons (n=20) were collected, cleaned of debris, and stored at 4°C until used. The teeth were bisected from the labial surface with a low-speed diamond saw under coolant water spray to obtain mesial and distal crown halves.

The primers of Clearfil SE Bond and Clearfil Protect Bond were applied by agitating the surface on one half and leaving it undisturbed on the other within the times recommended by the manufacturers. Xeno III and Adper Prompt, both being mixed into a single solution before application, were also applied in the same manner. A 35% orthophosphoric acid gel (Scotchbond Gel, 3M, St. Paul, Minn) was applied on the enamel surface for 30 and 60 seconds without agitation, while NRC was applied as it was to the other test materials. All experimental procedures were performed additionally on intact permanent enamel to enable morphological comparisons between the enamel of 2 dentitions. While the self-etch systems were applied on permanent enamel as they were on primary enamel, the phosphoric acid etching time on permanent tooth samples was reduced to 15 and 30 seconds. Except for phosphoric acid-treated samples, all enamel surfaces were thoroughly rinsed with acetone and ethanol to remove the uncured self-etching primers. Thereafter, specimens were dried chemically using hexamethyldisilazane (HMDS-Electron Microscopy Sciences, Fort Washington, Pa), mounted on aluminum stubs, sputter coated with gold-palladium (Balzers-SCd 050 sputter coater, Liechtenstein) and observed under a scanning electron microscope (JSM-6400 V, JEOL, Tokyo, Japan) at 20 kV of accelerating voltage.

RESULTS

PHOSPHORIC ACID

Phosphoric acid produced well-defined etching patterns on both primary and permanent intact enamel. Differential dissolutions of either prism cores or boundaries could be seen across the entire enamel surface both in primary and permanent teeth. On primary enamel, 60 seconds of etch time produced more pronounced etching patterns on the enamel surface than 30 seconds of etching time. Similarly, increased etching time resulted in more dissolution of the permanent tooth enamel (Figure 1).

CLEARFIL PROTECT BOND

Application of the primer in either treatment modes resulted in very poorly structured surface. Fine tracks that

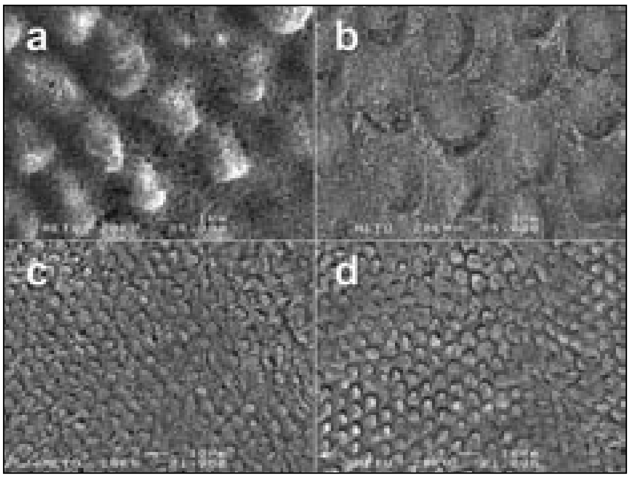

Figure 1. Appearance of primary and permanent enamel after treatment with 37% orthophosphoric acid: a. primary enamel (30 seconds); b. primary enamel (60 seconds); c. permanent enamel (15 seconds); and d. permanent enamel (30 seconds).

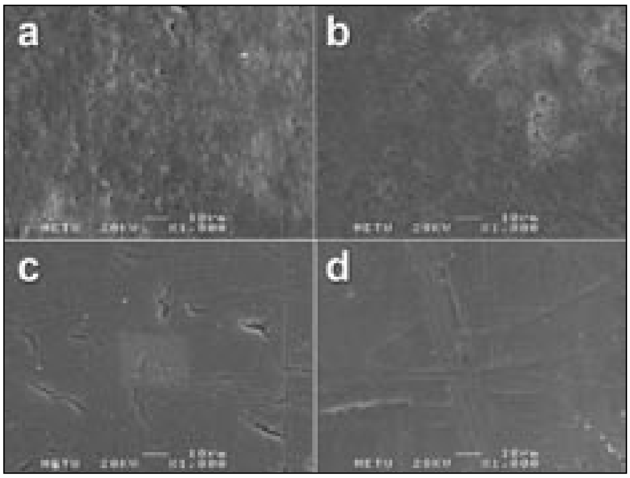

Figure 2. SEM micrographs of primary and permanent enamel after treatment with Clearfil Protect Bond: a. primary enamel (nonagitated); b. primary enamel (agitated); c. permanent enamel (nonagitated); and d. permanent enamel (agitated).

were observed along the enamel surface probably represented grooves that were created on unground enamel by either tooth brushing or polishing with the bristle brush before specimen preparation. The enamel surface was predominantly unetched, and only shallow fossae could occasionally be seen. Agitating the surface with the Clearfil Protect Bond primer merely improved dissolution of the enamel in primary and permanent teeth (Figure 2).

CLEARFIL SE BOND

When compared to the Clearfil Protect Bond primer, Clearfil SE Bond primer produced more dissolution on primary enamel than on permanent enamel. The etching patterns on both types of enamel, however, were still very weak (Figure 3).

NONRINSE CONDITIONER (NRC)

Unlike Clearfil SE and Clearfil Protect Bond groups, an overall increase in microporosity was evident along the entire aprismatic enamel surface treated with NRC. Etched appearance was more evident in the permanent teeth. Agitation of NRC on primary teeth resulted in a more roughened enamel surface (Figure 4).

XENO III

Agitation increased the etching efficacy of Xeno III in both primary and permanent teeth, while the etch patterns were still weak, showing local variations (Figure 5). Xeno II appeared to be more effective in primary enamel specimens.

ADPER PROMPT

When compared to primary enamel, Adper Prompt appeared to produce more dissolution on permanent enamel. The

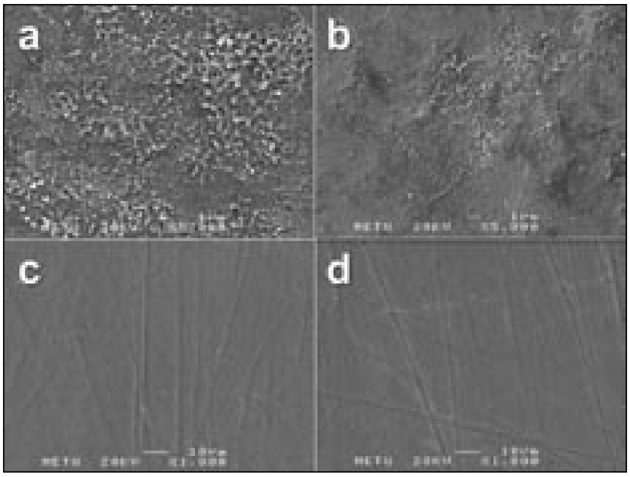

Figure 3. SEM micrographs of primary and permanent enamel after treatment with Clearfil SE Bond: a. primary enamel (nonagitated); b. primary enamel (agitated); c. permanent enamel (nonagitated); and d. permanent enamel (agitated).

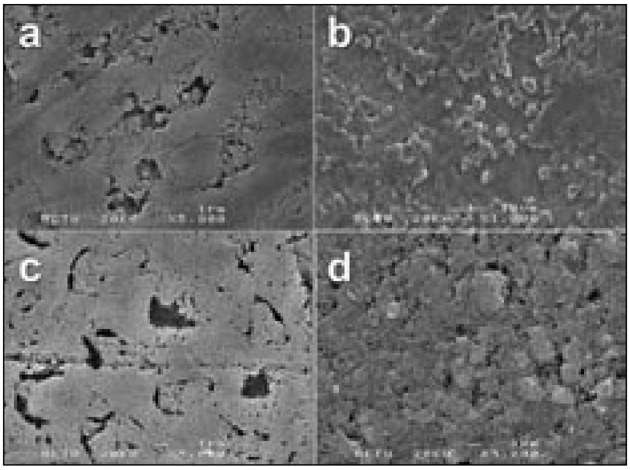

Figure 4. SEM micrographs of primary and permanent enamel after treatment with NRC: a. primary enamel (nonagitated); b. primary enamel (agitated); c. permanent enamel (nonagitated); and d. permanent enamel (agitated).

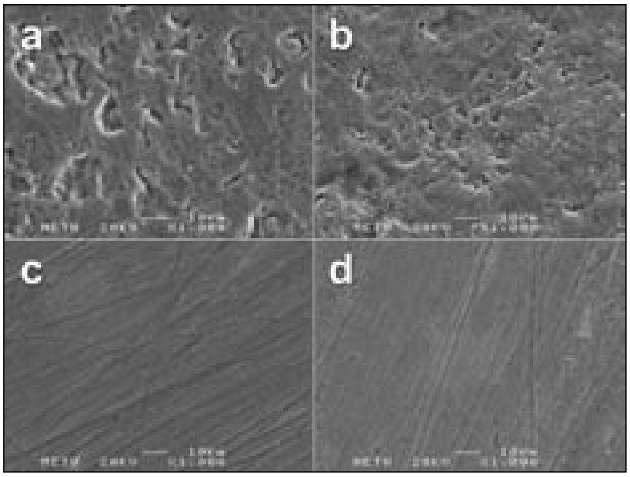

Figure 5. SEM micrographs of primary and permanent enamel after treatment with XENO: a. primary enamel (nonagitated); b. primary enamel (agitated); c. permanent enamel (nonagitated); and d. permanent enamel (agitated).

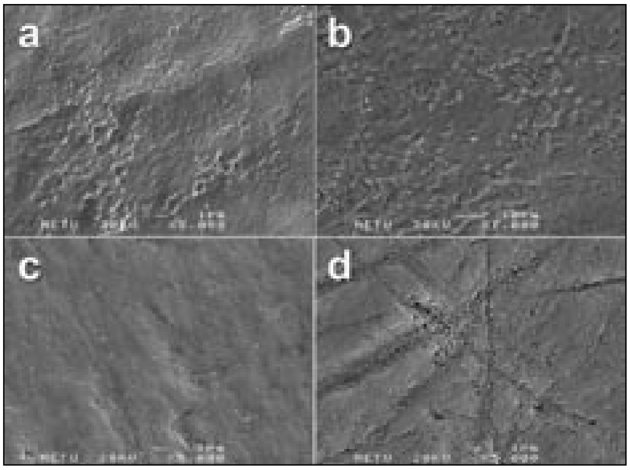

Figure 6. SEM micrographs of primary and permanent enamel after treatment with Adper Prompt: a. primary enamel (nonagitated); b. primary enamel (agitated); c. permanent enamel (nonagitated); and d. permanent enamel (agitated).

etching patterns in both agitated and nonagitated samples, however, were weak. Agitation of the primer had little influence in terms of enamel dissolution in primary enamel samples (Figure 6).

DISCUSSION

Studies on the adhesion of self-etching primers on enamel are recent, and results are not as consistent as those reported for the adhesion of the same products to dentin. Some authors have reported that the bond strength of self-etching primers is inferior to that obtained with adhesive systems, which utilize orthophosphoric acid as a surface conditioner.[13-15] Conversely, other studies that tested composite-to-enamel bond strength with self-etching adhesive systems have reported values as high as 20 to 30 MPa,[5,6,16] being in the same range as those reported on phosphoric acid-etched enamel.

The main purpose of the present study was to evaluate ultramorphological differences created by commercially available self-etch systems on unground human primary and permanent enamel. Miyazaki et al[8] reported higher bond strength to enamel when the specimens were prepared by agitating the primer on the surface. Nevertheless, the authors also concluded that the mineral component and the mechanical properties of adhesives may also play important roles in the determination of bond strength.

The degree of depth of enamel surface removed during the etching procedure depends on the type and concentration of the acid, the duration of etching, and the surface's chemical composition. When applied on enamel, phosphoric acid causes a selective dissolution of either enamel prism cores or boundaries and creates microporosity on the enamel surface ranging in depth from 5 to 50 μm.1 Unlike phosphoric acid conditioning, the action of self-etch systems on permanent tooth enamel has been reported to produce ill-defined surface structures.[5,6,17]

The present study's results corroborate those of previous findings, while demonstrating similar results in primary tooth unground enamel, except for Adper Prompt and Xeno III, which have not been reported previously. The shallow, undefined etching pattern is considered to be a result of deficient penetration of the self-etching primer into the enamel surface.[6] A recent study, however, demonstrated that resin-to-enamel bonding with self-etching primers is based on an inter- and intracrystallite hybridization of the enamel rather than dissolution and resin-tag formation. The authors described their findings as "nanoretentive" interlocking.[18]

The intact enamel surface is prismless and hypermineralized and contains more inorganic material when compared to ground enamel.[16] The prismless layer of enamel is thicker in primary teeth. This could be one of the reasons why self-etch systems create more shallow-etch patterns. Interestingly, self-etch systems with relatively higher pH (primers of Clearfil SE Bond and Clearfil Protect Bond) resulted in more dissolution on primary tooth enamel, while those with a lower pH (Adper Prompt) dissolved permanent tooth enamel more effectively. To complement these ultra-

Table 1. Composition of Self Etch Systems

Product	Composition		pH
XenoIII	**Liquid A** HEMA Purified water Ethanolurethane dimethacrylate resin Butylated hydroxytoluene Highly dispersed silicon dioxide	**Liquid B** Phosphoric acid-modified polymethacrylate resin Monofluoro phosphazene-modified methacrylate resin Urethane dimethacrylate resin Butyleted hydroxitoluene Ethyl 4 dimethylaminobenzoate	1.0 (mixed)
Adper Prompt L-Pop	**Liquid 1** Methacrylated phosphoric esters Bis-GMA* Initiators based on camphorquinone Stabilizers	**Liquid 2** Water HEMA† Polyalkenoic acid Stabilizers	0.8 (mixed)
Clearfil SE Bond	**Primer** MDP‡ HEMA† Hydrophilic dimethacrylate	**Adhesive** MDP, Bis-GMA, HEMA Silanated colloidal silica	Primer 1.9
Clearfil Protect Bond	MDP, MDPB§, HEMA water, hydrophilic dimethacrylate	MDP, Bis-GMA HEMA, microfiller, surface-treated sodium fluoride	Primer 1.9
NRC	Iatonic acid, maleic acid, carboxylic acid, water, solvent		1.2

**Bisphenol A dimethacrylate.*
†2-hydroxyethyl metachrylate.
†10-methacryloyloxydecyldihydrogen phosphate.
§Methacryloyloxydodecyl-pyridinium bromide.

morphological data with quantitative comparisons, further studies on enamel dissolution are required.

Morphological features of enamel surfaces treated with Clearfil SE bond and Clearfil Protect Bond were almost identical. The enamel surface showed a slight increase in porosity without any marked demineralization through the depth of enamel structure. In fact, the only reported difference between Clearfil SE bond and Clearfil Protect Bond primers is the incorporation of MDPB monomer in the Clearfil Protect Bond primer. Unlike the acidic MDP (10-methacryloyloxydecyl dihydrogen phosphate) monomer, which has a phosphate residue, the MDPB monomer has no etching ability but is claimed to possess antibacterial properties. The acidic resin monomers in Adper Prompt consist of methacrylated phosphoric acid mono- and diesters, in which 1 or 2 hydrogen atoms of orthophosphoric acid are replaced with at least one methacryloxy group. Containing a mixture of the more acidic monoesters and less acidic diesters, Adper Prompt has quite a low pH. On the other hand, Clearfil SE Bond contains the phosphoric acid monoester MDP has a higher pH and produced a substantially weaker etch pattern on enamel. NRC containing itaconic acid and maleic acid has a pH of 1.2 and produced moderately definitive etching effect.

CONCLUSIONS

Based on this study's results, the following conclusions can be made:

1. Agitation of the tested self-etching systems may result in more pronounced etching patterns on intact primary and permanent enamel. The etching effect, however, appears to be dependent on the material used.
2. When compared to phosphoric acid, NRC produced weaker etching patterns in both primary and permanent enamel.
3. The ultramorphological data obtained in this study needs to be supported with future quantitative studies on enamel dissolution as well as bond strength and microleakage tests.

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