Original Article

Effects of a Nonrinse Conditioner and 17% Ethylenediaminetetraacetic Acid on the Etch Pattern of Intact Human Permanent Enamel

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Abstract: The purpose of this study was to evaluate the effects of 2 new acid-etching solutions, nonrinse conditioner (NRC) and 17% ethylenediaminetetraacetic acid (EDTA), on enamel surface morphology, and to compare the new solutions with traditional 37% phosphoric acid. The effect of prolonged etching time was also investigated. The buccal surfaces of 80 extracted third molars were etched with one of the 3 acids for 15, 30, or 60 seconds. The central regions of the specimens were examined with a scanning electron microscope. Shorter etching time with phosphoric acid resulted in a relatively smooth enamel surface compared with longer treatments. Irrespective of treatment time, NRC produced an aprismatic etch pattern, which suggested a potentially retentive morphological character. EDTA treatment had the least effect of all etchants tested. (*Angle Orthod* 2000;70:22–27.)

Key Words: Acid etching, Compomer, NRC, EDTA

INTRODUCTION

Since the introduction of the acid etch technique by Buonocore,[1] the bonding of orthodontic attachments to acid-etched enamel has become an accepted clinical procedure.[2] The effects of phosphoric acid concentration and duration of etching on enamel surface morphology and bond strength of orthodontic resins have been investigated by several authors.[3–6] Because successful clinical bonding can be obtained by a shear bond strength of 6 to 8 Megapascals,[7] conventional enamel etching with 37% phosphoric acid has been reduced from 60 seconds to 15 seconds, with minor effect on bond strength.[8] However, the use of composites for bracket attachment has a number of disadvantages, including enamel loss that can occur during prophylaxis,[9] acid etching,[10] and debonding.[11] Therefore, considerable effort has been made to develop bonding materials that help preserve dental enamel.

Glass ionomer cements, which possess many favorable characteristics, have historically been applied directly to enamel without acid etching.[12] However, results of both laboratory tests and clinical performance evaluations indicate that conventional glass ionomer cements are not universally recommended for the bonding of brackets because of their inferior bond strength.[13–15] Currently, however, there is a consensus of opinion forming that favors the use of a new generation of hybrid materials that contain both resin and glass ionomer.[12] One of these materials is the light-activated product Dyract Orthodontic (DeTrey Dentsply, Konstanz, Germany). This material belongs to a new class of materials called polyacid-modified resin composites, or compomers. Although some confusion still exists surrounding the exact formulations of compomers, a clear classification for such hybrid materials has been introduced.[16] Accordingly, the terms ‘‘polyacid-modified resin composite’’ and ‘‘compomer’’ have been reserved for materials that contain essential components of a glass ionomer cement but at levels that are insufficient to produce an acid-base reaction (the main setting mechanism of conventional glass ionomers) in the dark. Curing of the material depends solely on photopolymerization, whereas the acid-base reaction, initiated by water from the oral environment, is responsible for the fluoride release.[17] Resin-modified glass ionomer cements (such as Fuji Ortho LC, GC America Inc, Chicago, Ill) retain a significant acid-base reaction as part of their overall curing process, with initial hardening that depends on photoactivation.[16] Major compositional differences between these 2 classes of hybrid materials could therefore explain the adequate bond strength of Fuji Ortho LC with no enamel pretreatment,[18] whereas Dyract Ortho, predominantly a composite resin, requires enamel etching with phosphoric acid.[19,20]

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TABLE 1. Enamel Etching Treatments*

Group (n = 10)	Acid	Composition	Treatment Time, (s)	Rinsing Time, (s)
1	Total etch	37% phosphoric	15	20
2	Total etch	acid in	30	20
3	Total etch	silica gel	60	20
4	NRC	Itatonic acid	20	No rinsing,
5	NRC	Maleic acid	40	air-drying
6	NRC	Water	60	for 5 seconds
7	17% EDTA (pH 7.0)	EDTA (17 g), NaOH (9.25 mL),	30	20
8	17% EDTA (pH 7.0)	distilled water (100 mL)	60	20

* NRC indicates nonrinse conditioner; EDTA, ethylenediaminetetraacetic acid.

When a bond failure occurs, it is likely to be the result of moisture contamination of the bonding site.[21] Several in vitro and in vivo studies have demonstrated that moisture contamination of etched enamel greatly reduces bond strength.[21–24] To overcome this clinical problem, the manufacturer of Dyract Orthodontic has recently manufactured a nonrinse conditioning solution (NRC, DeTrey Dentsply, Konstanz, Germany) that they claim will etch the enamel without further rinsing.[25] Because the procedure does not require rinsing, the risk of saliva contamination, which may occur during change of cotton rolls after rinsing, is reduced.

The etching effect of ethylenediaminetetraacetic acid (EDTA), another enamel conditioner, has recently been evaluated on ground permanent human enamel.[26] The scanning electron microscope photographs obtained in this study revealed a smooth, wavelike, and reactive etched surface, while the integrity of enamel prisms was maintained.[26] However, the efficacy of EDTA for orthodontic purposes (unground enamel) remains to be substantiated.

The purpose of this study was to evaluate the morphology of unground permanent human enamel after etching with NRC and 17% EDTA solutions. In addition, the effect of prolonged conditioning time of the test solutions were compared with 37% phosphoric acid applied to unground enamel for 15, 30, and 60 seconds.

MATERIALS AND METHODS

Unerupted human third molars were extracted and stored in a 0.5% chloramine solution for no longer than 3 weeks prior to use. The crowns were sectioned from the roots with a diamond disc at the cementolabial enamel junction, and each crown was cut longitudinally in a mesiodistal direction. The buccal surfaces of the crowns were cleaned thoroughly with a rubber cup and a slurry of pumice and water, followed by rinsing with water spray and drying with compressed air. A total of 80 buccal enamel surfaces were prepared and assigned to 1 of 8 groups with 10 specimens per group (Table 1).

The enamel surfaces of the teeth in groups 1, 2, and 3 were etched with 37% phosphoric acid gel (Total Etch, Vivadent, Schaan, Liechtenstein) for 15 seconds, 30 seconds, and 60 seconds, respectively. Following rinsing for 20 seconds, the enamel surfaces were dried with oil-free compressed air for 10 seconds. Samples in groups 4 to 6 were treated with NRC, a nonrinse conditioner containing organic acids and monomers in an aqueous base.[25] Itatonic acid, which functions as the primer, copolymerizes with the subsequently applied bonding agent and its carboxylic groups adhere to calcium of the tooth surface, while maleic acid conditions and cleanses the tooth surfaces. After 20 seconds of treatment, the surface is air-dried for 5 seconds, after which the remnants of NRC are incorporated into the bonding agent as filler particles. The samples in group 4 were treated for 20 seconds, as recommended by the manufacturer. In groups 5 and 6, enamel surfaces were treated for 40 seconds and 60 seconds, respectively, in order to observe the effect of treatment time on surface morphology. The 17% EDTA solution at pH 7.0 was prepared in laboratory conditions.[27] The composition of the test solution is shown in Table 1. Because EDTA is a weak acid,[28] the samples in groups 7 and 8 were treated for 30 seconds and 60 seconds, respectively, followed by rinsing and drying, as for the samples treated with phosphoric acid (Table 1).

All samples were mounted in metal stubs and coated with gold in an E5200 Auto Sputter Coater (BIO-RAD, Polaron Equipment Ltd, Watford, England) as a preparation for SEM. Each sample was then analyzed in a scanning electron microscope (JEOL 6400, Tokyo, Japan) with an accelerating voltage of 20.0 Kv. A thorough scan of the area was performed to evaluate the general morphological characteristics of the specimens and to allow the operator to select the most representative fields for the micrographs. Although the etching patterns were sometimes too heterogeneous, even between adjacent areas, all samples within each group were similar.

According to Silverstone et al[29] and Galil and Wright,[30] there are 5 types of etching patterns, and these were used as diagnostic criteria:

- Type 1: Preferential dissolution of the prism cores, resulting in a honeycomblike appearance.
- Type 2: Preferential dissolution of the prism peripheries, giving a cobblestonelike appearance.

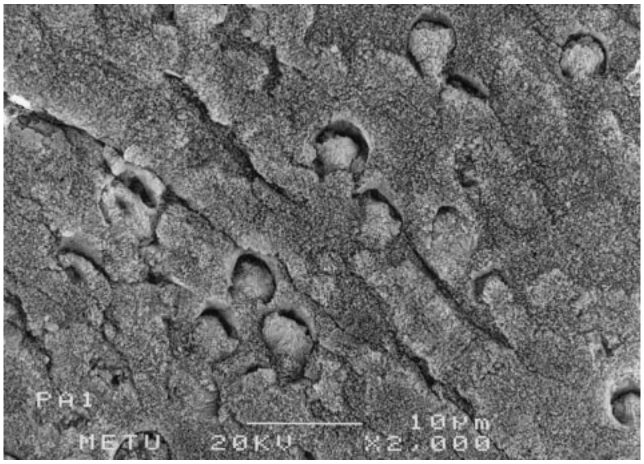

FIGURE 1. Enamel pattern observed following treatment with 37% phosphoric acid for 15 seconds. Note nonuniform and flat type 2 pattern associated with fine granulation of the enamel surface.

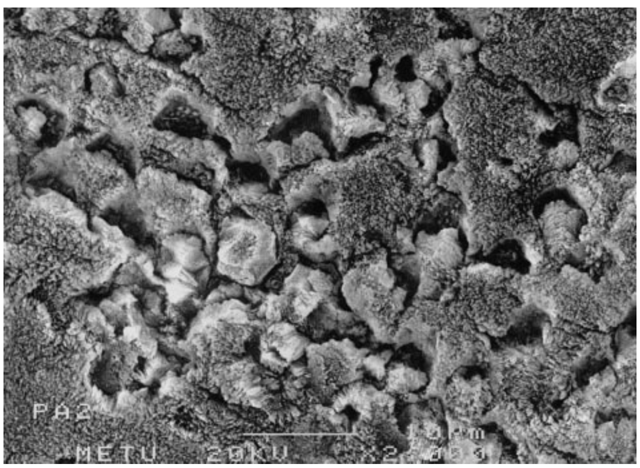

FIGURE 2. Enamel pattern observed following treatment with 37% phosphoric acid for 30 seconds. Note predominantly type 2 pattern of enamel surface with numerous holes characteristic of the type 4 pattern.

- Type 3: A mixture of type 1 and type 2 patterns.
- Type 4: Pitted enamel surfaces as well as structures that look like unfinished puzzles, maps, or networks.
- Type 5: Flat, smooth surfaces.

RESULTS

The intact enamel surfaces etched with different acids had different surface morphologies. In general, the 30-second and 60-second treatments with 37% phosphoric acid affected the prism cores to varying degrees, leaving a rough enamel surface. The 15-second application of phosphoric acid resulted in only partial removal of the enamel apatite crystals in which a flat, but often nonuniform, type 2 etching pattern was evident (Figure 1). With the 30-second treatment, phosphoric acid formed a rougher surface in which the type 2 etching pattern of the enamel was more

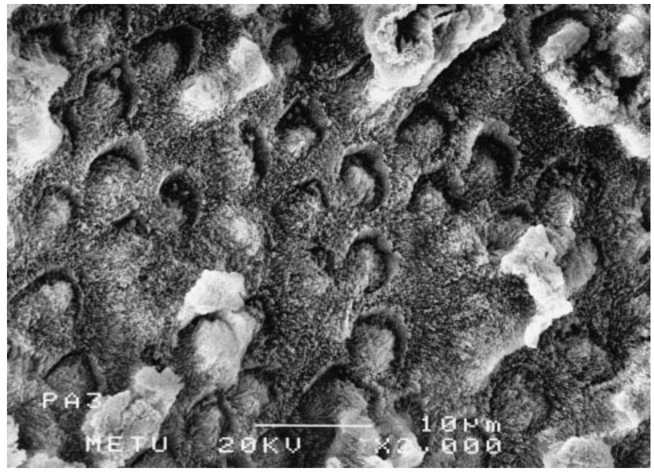

FIGURE 3. Enamel pattern observed following treatment with 37% phosphoric acid for 60 seconds. Note predominantly flat surface associated with aprismatic etching (maplike formations) characteristic of the type 4 pattern.

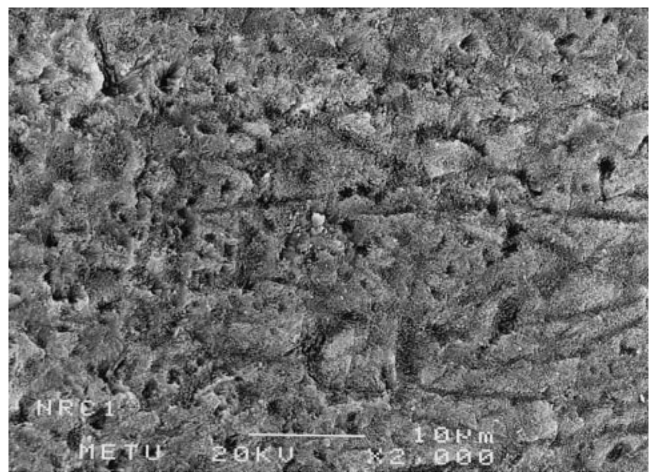

FIGURE 4. Enamel pattern observed following treatment with nonrinse conditioner for 20 seconds. Note aprismatic etching with fine surface roughening and pitted enamel surfaces characteristic of the type 4 pattern.

frequently observed (Figure 2). In addition, pitted surfaces and roughening of the crystallites were noted. The 60-second phosphoric acid treatment resulted in a very rough enamel surface with a more uniform type 2 etching pattern (Figure 3). To a lesser extent, a nonuniform distribution of aprismatic etching sites was observed.

For the NRC groups, the most smoothly etched surface was obtained with the 20-second treatment, which produced only superficial demineralization of the enamel (Figure 4). No loss of enamel prisms was evident, although the homogenous porosity of the enamel associated with a pattern of generalized pitting suggested a potentially retentive surface (Figure 4). The 40-second and 60-second treatments with NRC resulted in rougher enamel textures (type 4 and

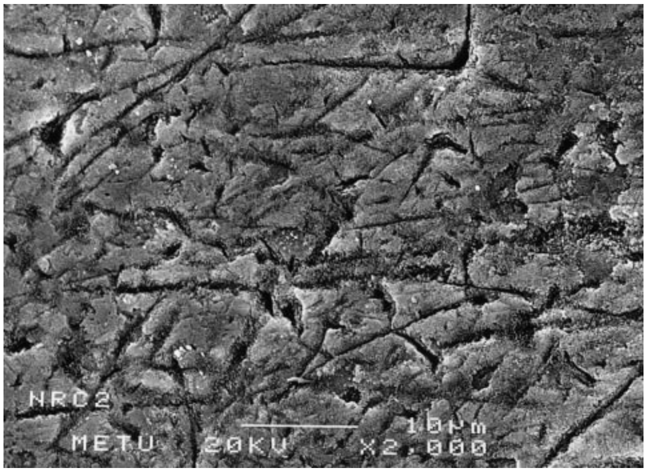

FIGURE 5. Enamel pattern observed following treatment with nonrinse conditioner for 40 seconds. Note aprismatic etching with increased surface roughening characteristic of type 4 and type 5 patterns.

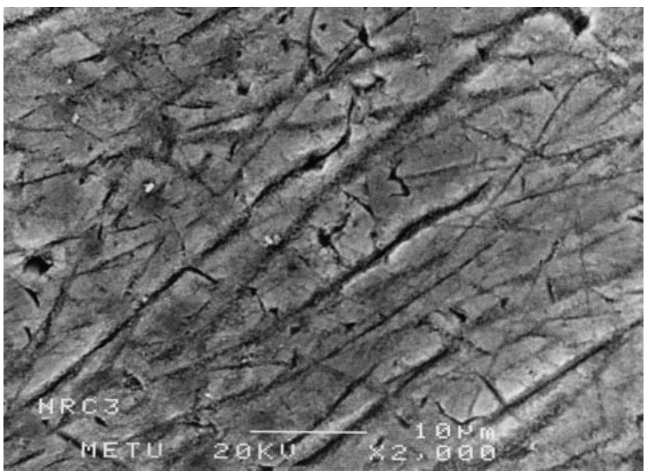

FIGURE 6. Enamel pattern observed following treatment with nonrinse conditioner for 60 seconds. Note aprismatic etching with increased surface roughening characteristic of type 4 and type 5 patterns. No significant change is evident when this surface is compared with the surface treated for 40 seconds.

5 patterns), but the integrity of the prisms was maintained (Figures 5 and 6).

Etching with EDTA for 30 seconds did not significantly alter the intact enamel surface (type 5 pattern, Figure 7). Only a smooth surface associated with nonuniform shallow pittings was visible. Similar results were observed for the 60-second treatment group (type 5 pattern, Figure 8).

DISCUSSION

The present investigation demonstrated a great variation in the morphology of etched enamel surfaces. Using the same etching procedures within a group, etching patterns were sometimes observed to vary between adjacent areas of the same tooth. Nevertheless, all the samples within a group were found to be similar. For this reason, it appeared to be impossible to quantitate the extent of surface irregularity, as previously described.[31] This valuation is in agreement with the findings of Denys and Retief.[32]

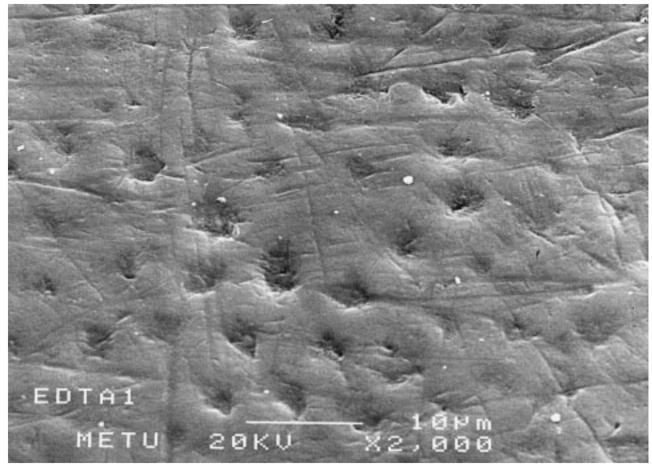

FIGURE 7. Enamel pattern observed following treatment with ethylenediaminetetraacetic acid for 30 seconds. Note flat, smooth surfaces with shallow pitting characteristic of the type 5 pattern.

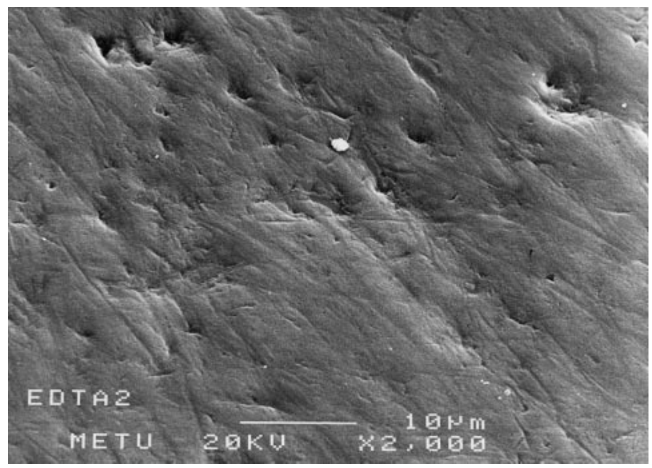

FIGURE 8. Intact enamel surface treated with 17% ethylenediaminetetraacetic acid for 60 seconds. Prolonged etching time has not significantly affected the surface morphology.

Based on the investigations of Silverstone,[33] Retief,[34] and others, acid solutions in concentrations of 20% to 50% applied for 1 minute to 2 minutes are thought to produce the most retentive conditions, and they have been recommended for clinical use for many years. This recommendation persists despite the fact that several investigators have shown no significant difference in bond strength with an exposure time of less than 30 seconds. Today, there seems to be a growing preference for softer etching procedures.[4,5,35,36]

The results of this study of enamel surface morphology achieved with phosphoric acid treatment are in agreement with previous studies[37,38] that demonstrated increased enamel surface roughness with prolonged etching times. Increased surface roughness renders the enamel more retentive and produces higher bond strength.[37,38] However, this may not be desirable from a clinical standpoint, because bond strength produced with an etching time as low as 15 seconds is still greater than that required for successful orthodontic bonding.[39] Additionally, even this relatively short etching period results in substantial irreversible loss of superficial enamel and crystallites, as shown in the present investigation and in previous studies.[3–5]

Compared with phosphoric acid, NRC produces an etch pattern that is far less destructive to the unground enamel surface. Even with the longest application time for NRC, no visible damage to the enamel crystals was detectable, whereas nonuniform loss of the prisms was evident with the shortest treatment time with phosphoric acid. The etching patterns shown in Figure 4 may well suggest that adequate and more conservative etching of the enamel surface can be obtained following the 20-second NRC treatment, without a need for prolonged etching time. The retentive type 1 and type 2 etch patterns were not present in any of the NRC treatment regimens. However, in the context of bonding, the rheological properties of the resin and the increase in surface area may be more significant than the depth of resin penetration.[35] This can be inferred from previous laboratory investigation, which suggested that a change of depth in enamel after a 15-second phosphoric acid application should be less than that after a 60-second application, whereas bond strengths should not be statistically different.[8,38,40] No previous study has investigated the effect of NRC on unground permanent enamel, although the initial data obtained in this investigation lends promising support for further bond strength studies with orthodontic compomers, and possibly with other resin adhesive systems. From a clinical standpoint, the use of a nonrinse etchant could be desirable because the bond strength of resin-based orthodontic adhesives has been shown to decrease significantly with moisture and saliva contamination.[24]

With regard to EDTA, a uniform and smooth type 2 etching pattern shown by Blomlöf et al[26] on ground enamel was in stark contrast to the type 5 pattern found on unground surfaces in the present study. The chemical and micromorphological features of unground enamel may account for this difference.[35] Also, the concentration used in the present study may be too low to obtain a desirable etching effect.[26] Within the limitations of this study, a 17% EDTA solution was not found suitable for etching unground enamel, because it offers no advantage over phosphoric acid or NRC in either efficiency or application time.

CONCLUSIONS

Regardless of treatment time, etching with 37% phosphoric acid results in irreversible damage of the enamel surface. Reduced etching time creates a smoother enamel surface with less absolute enamel loss. On the basis of present observations and previous bond strength studies, 15-second etching time with 37% phosphoric acid is suitable for producing acceptable bracket bond strength while minimizing permanent enamel loss.

NRC treatment produces a smooth yet ‘‘adequately rough’’ enamel surface for bonding without a need for prolonged etching time. Because these alterations are limited to the surface, with no damage to the enamel prisms, treating enamel with NRC may be advisable if brackets are to be bonded with compomers.

Enamel etching with 17% EDTA cannot be recommended for orthodontic purposes.

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