

# The Effect of two different etching modalities (EDTA and Phosphoric Acid ) on texture of enamel and dentin

Methal F. Shakir BDS, M.Sc, Operative Dentistry

AL-Yarmook universal college, Department of operative Dentistry , Iraq.

## خلاصة البحث:

الهدف من هذه الدراسة هو فحص سطح ميناء و عاج السن المعامل بنوعين مختلفين من المواد المخرشة وهي حامض الفسفوريك وحامض الاثيلين داي امين تترا استك (EDTA) باستعمال المايكروسكوب الالكتروني الماسح وبتكبير ١٠٠٠ و ٤٠٠٠ .

بدا العمل بتجميع و تنظيف (١٢) سن من نوع الضواحك للفك السفلي و من تم تقطيعها الى شرائح عريضة للحصول على الطبقة السطحية من العاج ومن تم صقلت بورق السيليكون و قسمت الى ثلاثة مجاميع و كما يلي:

- المجموعة الاولى (١): استخدمت كعينة مقارنة بدون معاملتها باي نوع من الحوامض المخرشة
- المجموعة الثانية (٢): وفيها سطح الميناء و العاج عومل بحامض الفسفوريك جل بتركيز ٣٧% و لمدة ١٥ ثانية لغرض التخريش.
- المجموعة الثالثة (٣): وفيها سطح الميناء و العاج عومل بحامض الاثيلين داي امين تترا استك جل و بتركيز ٢٤% لمدة دقيقة واحدة.

ثم تم طلاء العينات بطبقة الذهب باستعمال الطبقة الايونية ثم ثبتت العينات على مسند باستعمال لاصق ثنائي الطبقة. ثم تم فحص سطوح الميناء و العاج لكل العينات باستعمال المايكروسكوب الالكتروني الماسح. لقد تبين من نتائج الفحص:

- (١) ان حامض الفسفوريك هو افضل من حامض الاثيلين داي امين تترا استك في تخريش مادة الميناء.
- (٢) ليس هنالك فرق واضح في ازالة المادة السطحية (smear layer) لسطح العاج لكل من الحامضين المخرشين.

## كلمات المفتاح:

اثيلين داي امين تترا استك، حامض الفسفوريك، المايكروسكوب الالكتروني الماسح، تخريش

## Abstract:

The purpose of this study was to investigate enamel and dentin surfaces that treated with different etching modalities - ethylynediaminetetraacetic acid conditioning (EDTA) and phosphoric acid conditioning using scanning electron microscop (X1000,X4000) .

**Method and materials:** Twelve sound human upper first premolar teeth, recently extracted for orthodontic purpose were selected . The occlusal surfaces of the teeth were sectioned horizontally so that the first layer of dentin was obtained. Then the teeth were stored in an incubator at 37°C for one hour .They were randomly divided into 3 groups,(each group containing 4 teeth),and categorized as following:

Sample 1: used as a control without etching

Sample 2: enamel and dentin were etched with phosphoric acid gel 37% for 15 sec.

Sample 3: enamel and dentin were etched with EDTA 24% gel for 1 minutes.

The specimens were gold sputter coated using an ion coating (E 5000 M S.E.M coater) and fixed with double layer adhesion on the stage . Then the dentin surface of all specimens were examined with a Scanning Electron Microscope SEM (Phillips SEM 510) .

**Result:** There was no differences between the two etching modalities for dentin etching. While phosphoric acid was superior for enamel etching ,

**Conclusion:** Although the result shows that EDTA was superior for enamel etch and there was no difference between the two modalities for dentine etch, more studies have been suggested with higher magnification (x10000) to see collagen fiber.

**Key words:**

EDTA, SEM, phosphoric acid, etching

**Introduction:**

The preparation of dental cavities inevitably produces a thin layer of smear that covers the dentin and enamel walls [1]. This smear layer, which is between [1] and 10  $\mu$ m in thickness, contains hydroxyapatite, denatured collagen, and remnants of cariogenic bacteria [2; 3]. If left untouched, it will weaken the bond strength between filling materials and the cavity wall [4]. Thus, etching of the cavity walls has been recommended since the mid 1950s to remove the smear layer.

The purpose of enamel etching is to remove smear layer and to create micro retentions by dissolving the prism centers, producing a honeycomb pattern that increases the area available for bonding. Dentin etching also aims at removing smear at the openings of the dentin tubuli, in addition to fibers of the peritubular and intertubular dentin [2].

Etchants such as phosphoric acid and ethylenediaminetetraacetic acid (EDTA) had been shown to remove most of the smear layer and leave the dentinal tubules opened. Additionally, these etchants had been shown to expose collagen fibrils to varying degrees on intertubular dentinas well as on the walls of dentinal tubules [5 ; 60]. In order to facilitate formation of a hybrid layer, etching is performed to remove or demineralize the smear layer and expose enough sound collagen fibrils to permit their envelopment and sealing of the dentin surface by resin. [7].

Traditionally, an aqueous gel preparation of 37% phosphoric acid at pH 1 has been recommended for etching.

However, this is a rather strong acid, and care must be taken not to damage the collagenous matrix of the dentin. Strong acids at low pH denature proteins such as collagen, which leads to a change in their dimensions, or even to fragmentation [8]. Such a sub-optimal surface interferes with subsequent resin infiltration and prevents the formation of a hybrid layer [9], although different bonding systems use the exposed collagen of varying degrees [10]. Nevertheless, adhesion between an etched collagen rich dentin surface and resin form a bonding system which is at least in part chemical [11, 12].

Experimental work on root surface conditioning has demonstrated that phosphoric acid has the potential for eroding the dentin surface, rather than exposing collagen selectively [13], it more or less dissolves the collagen fibers. Instead, a supersaturated (EDTA) solution at pH 7 has been found not only to remove hydroxyapatite selectively, but also to preserve the integrity of the collagen fibers [14]

**Method and Materials:**

Twelve sound human upper first premolar teeth, recently extracted for orthodontic purpose (the patient's age range from 13-20 years) of comparable size and shape were selected. The teeth then stored in deionized distilled water (DDW) in the refrigerator at 4°C until sample preparation [14]. The buccal and palatal cusps were cut by diamond cutting disk (with continuous cooling by distilled water spray) with the level of the occlusal pits and fissure and perpendicular with the long axis of the tooth. The occlusal surface of each tooth was ground against the flat wet surface of abrasive paper. At the end of this procedure, a flat, polished enamel and dentine surface (the first layer of dentin) was obtained. Then the teeth were stored in an incubator at 37°C for one hour. The samples were randomly divided into 3 groups, (each

group containing 4 teeth), and categorized - as following:

Sample 1: used as a control without etching

Sample 2: enamel and dentin were etched with phosphoric acid gel 37% for 15 sec.

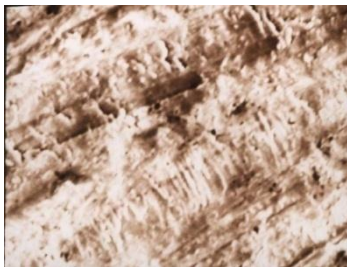
Sample 3: enamel and dentin were etched with EDTA 24% gel for 1 minute.

The specimens were gold sputter coated using an ion coating (E 5000 M S.E.M coater) and fixed with double layer adhesion on the stage. Then the enamel and dentin surface of all specimens were examined with a Scanning Electron Microscope SEM (Phillips SEM 510).

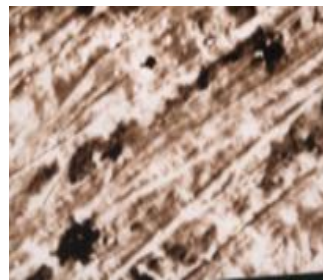
- **Result:**

- **Sample No. 1:** is considered a control specimen in which no etching material was used. Examination result showed that the smear layer covered both enamel and dentin. No protruding prism sheaths were notable on the surface of the enamel (Fig 1.a). Smear layer covers the opening of the dentin tubules (Fig. 1.b).
- **Sample No. 2:** in which both enamel and dentin were etched with Phosphoric acid. Examination results showed that enamel surface had wave-like appearance with protruding prism sheaths (Fig. 2.a). While dentin surface displayed patent dentin tubules (Fig. 2 b). At higher magnification (x4000), no definitely clear collagen fibers could be visualized (Fig 2 c).
- **Sample No. 3:** in which both enamel and dentin were etched with EDTA. The results of examination showed no trace of smear. The contour of enamel prisms

could not be seen clearly (Fig. 3 a). Dentin surface was also free from smear with a patent dentin tubules (Fig. 3 b). Higher magnification (x4000) showed no clear evidence of the presence of collagen fibers (Fig. 3 c).

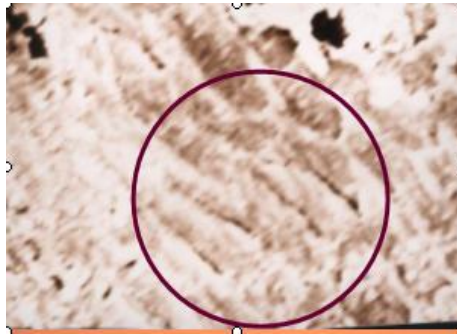


(Fig 1a)  
( Enamel)



(Fig 1b)  
( Dentine)

No etching material was used

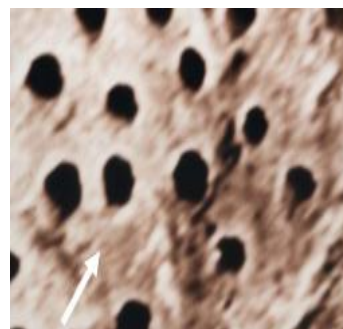


(Fig 2a)

( enamel )  
Etching with phosphoric

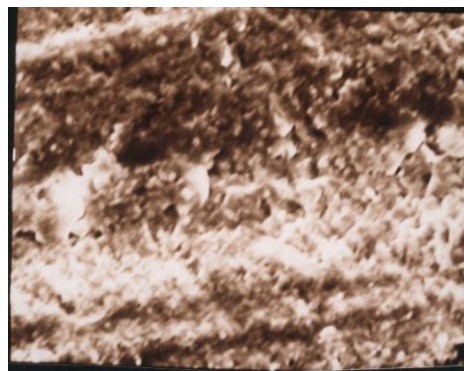


(Fig 2b)  
X1000

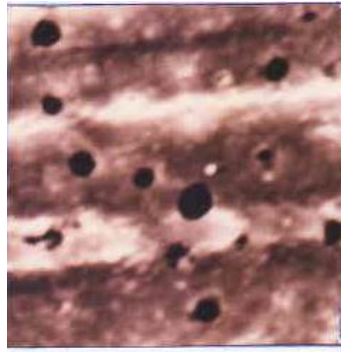


( Fig 2c)  
X 4000

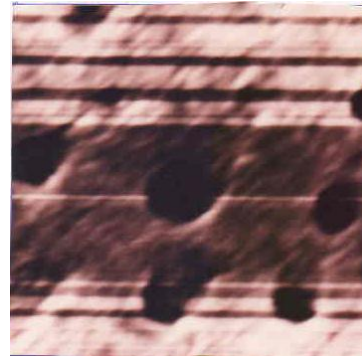
( Dentine)  
Etching with phosphoric acid



(Fig 3a)  
( Enamel )  
Etching with EDTA



(Fig 3b)  
X 1000



(Fig 3c )  
X4000

( Dentine )  
Etching with EDTA

### Discussion :

Upper first premolar chronologically young teeth (extracted for orthodontic treatment) were used in this study, the age of the patients ranged between 12-20 years. These teeth were with healthy periodontal. The occlusal surfaces were prepared with 600 grit abrasive paper [15] to be precise to expose the first layer of dentin after removal of all traces of enamel in order to get the same number of dentine tubules. [16].

The purpose of enamel etching is to remove smear and to create micromechanical retention, thereby increasing the area available for bonding. Etching of dentin surface also removes smear and opens dentin tubules. However, ideally it should also expose collagen fibers of the peritubular and intertubular dentin [17; 10].

The exposed collagen, in which some hydroxyapatite still remains, will form a hybrid layer with resin component (Primer) of restorative composite material upon polymerization. The hybrid layer will bind to the resin-based restorative material in subsequent steps through the action of an adhesive [18].

The preservation of collagen fibers in the dentin surface thus appears to be imperative to avoid jeopardizing bonding at subsequent step [15].

In this study, the sample that was etched with phosphoric acid showed by using SEM (X1000, X4000) that the enamel surface has wave-like appearance where the outline and full contour of the enamel prisms could be seen. While the sample that was etched with EDTA showed that the outline and full contour of enamel prisms could not be clearly seen and this could be explained that the effect of EDTA was not enough to clear the outline of enamel prisms. This result agreed with findings of Johan, et al, 1999, and this probably related to the less powerful effect of EDTA on the enamel in comparison with phosphoric acid. Regarding the effect of etching on dentin, there was no evident difference in smear removing ability between the different treatment procedures and both of them showed patent dentin tubules by the available magnifications (up to x4000) that was applied. However, at higher magnifications by SEM (at x10000) the collagen fibers could be identified

as have been achieved by Johan, et al, 1999 confirming the effect of EDTA (Fig. 2.5 & Fig. 2.6) where it is not feasible at the time being.

### Conclusion:

With this study we conclude that:

- 1) Phosphoric acid is superior than EDTA for enamel etching
- 2) No differences between the two modalities for dentine etching
- 3) More studies have been suggested with higher magnification (x10000) to see the effect of each modality on collagen fibers.

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