# The effects of different phosphoric acid concentrations on surface enamel

By Wolfgang Carstensen, DMD

Etching of dental enamel surfaces with phosphoric acid is an accepted and widely used technique, not only in orthodontics, but also in many other fields of dentistry. Buonocore[1] first demonstrated that the adhesion of acrylic materials to enamel increased after pretreatment of the teeth with phosphoric acid. Based on the investigations of Silverstone,[2] Retief,[3] and others, acid solutions in concentrations of 20-50% applied for 1-2 minutes were found to produce the most retentive conditions, and they were recommended for clinical use for many years. However, numerous subsequent studies have shown that a reduction of acid concentration and/or application time might not have adverse effects on the bonding process, and today there seems to be a tendency to softer etching procedures.[4]

Soetopo et al.[5] measured the tensile bond strengths after etching with 2-60% phosphoric acid solutions. The 16% acid obtained the highest bond strength, but the values for 2% were similar to those for 40%. Zidan and Hill[6] also found no significant difference in tensile bond strength after the 1-minute application of 2%, 5%, and 35% phosphoric acid. The loss of enamel, however, was considerably higher at 35% than at 2%. Gottlieb et al.[7] examined the tensile bond strength after etching with 10-60% phosphoric acid solutions and determined no significant differences. Legler et al.[8] measured the shear bond strength after etching with 37%, 15%, and 5% phosphoric acid and ascertained that the acid concentration had no significant effect on the bond strength values. The studies of Barkmeier et al.,[9] Bryant et al.,[10] and Weissenberg and Diedrich[11] also have shown that a phosphoric acid concentration of 5% is probably appropriate to achieve sufficient bond strength.

The aim of the present investigation was to evaluate the morphological effects of various phosphoric acid concentrations on dental enamal surfaces.

**Abstract**

The purpose of this study was to evaluate the effects of different acid concentrations on the enamel surface morphology. The buccal surfaces of 25 extracted premolars from young patients were etched with 40%, 20%, 10%, 5%, and 2% phosphoric acid solutions for 60 seconds. The specimens were examined with a scanning electron microscope in the occlusal, central, and cervical regions. A great variation of the etching patterns was observed in almost all test groups. The extent of the appearance of prism outlines was smaller in the cervical region and at lower acid concentrations. The advantages and disadvantages of the use of lower concentrated acids are discussed.

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**Key Words**

Bonding • Dental enamel • Acid etching

**Figure 1**
**40%, Central 'Cobblestone' appearance, pronounced type 2 pattern (bar = 10 μm)**

**Figure 2**
**40%, Occlusal Nonuniform and flat type 2 pattern (bar = 10 μm)**

Figure 1

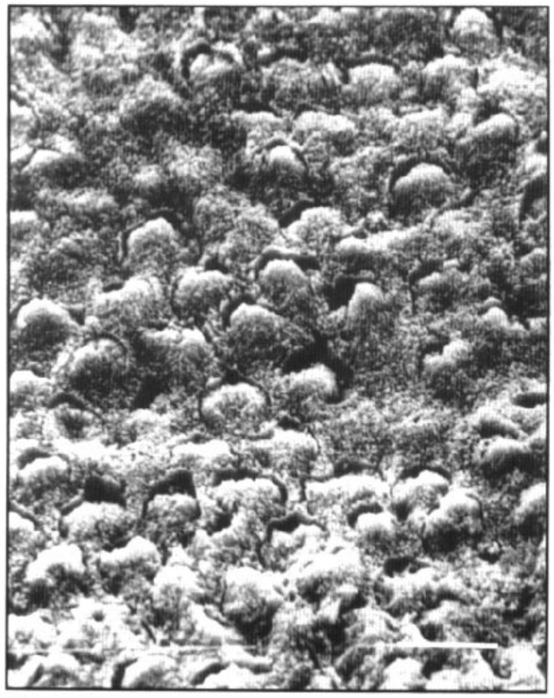

Figure 2

## Materials and methods

The material consisted of sound maxillary and mandibular premolars from young patients, 10 to 17 years of age. The teeth had been extracted for orthodontic reasons, and were stored in 35% ethanol (for about 2 to 6 weeks). Before the start of the experiments, the teeth were rinsed for 24 hours under running water. The crowns were sectioned from the roots with a diamond disc at the labial cemento-enamel-junction, and each crown was cut longitudinally in a mesio-distal 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 28 buccal enamel surfaces were prepared.

The five acid concentrations used were 40%, 20%, 10%, 5%, and 2%. All solutions were obtained by diluting of liquid 85% phosphoric acid and measured as weight percentages. The application time was 60 seconds. Each of the study groups contained 5 specimens. Three teeth were prepared in the same way, but not etched, and served as controls to facilitate the differentiation of etching patterns and normal tooth structures.

The chosen acid solutions were generously spread over the prepared enamel surfaces with a minisponge, and agitated slightly during the application. After etching, the teeth were rinsed with water spray for 30 seconds and dried. All specimens were mounted on aluminum stubs and prepared for SEM by sputtering with gold in a high vacuum evaporator (Balzers Hochvakuum GmbH, 6200 Wiesbaden-Nordenstadt, Germany). They were examined in a Philips SEM 501 scanning electron microscope, operated at 15 kV with a beam-specimen angle of about 55°. The buccal surfaces of the etched teeth and of the controls were inspected carefully at three sites: about 2 mm from the cusp tip (occlusal), in the middle (central), and about 2 mm from the cemento-enamel-junction (cervical). A total of 108 photographs were taken, at least one of each site of each tooth. Magnifications ranged between 20 and 10 000 times. The attempt was made to obtain photographs that were typical of the appearance of the enamel at that site, but sometimes the etching patterns were too heterogeneous, even between directly adjacent areas. The film material used was Ilford HP 5 Rollfilm.

According to Silverstone et al.[12] and Galil and Wright[13] the etching patterns were related to five types:

Type 1: preferential dissolution of the prism cores resulting in a "honeycomb" appearance.

Type 2: preferential dissolution of the prism peripheries giving a "cobblestone" appearance.

Type 3: a mixture of type 1 and 2 patterns.

Type 4: pitted enamel surfaces as well as structures which look like unfinished puzzles, maps, or network.

Type 5: flat, smooth surfaces.

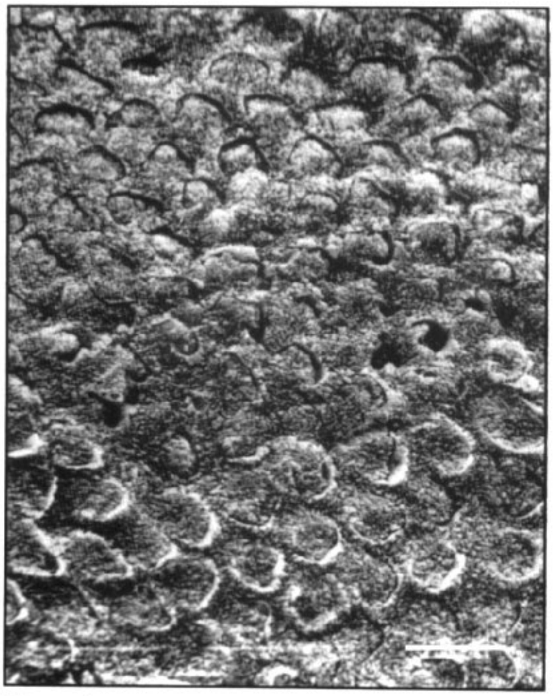

Figure 3

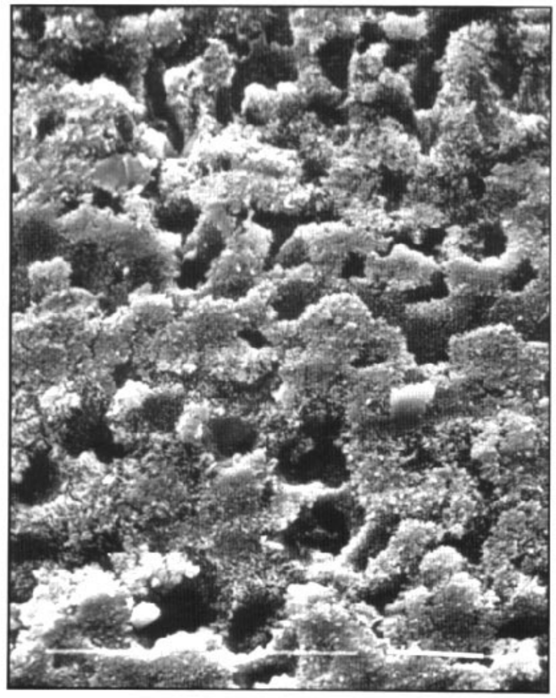

Figure 4

**Figure 3**
**40%, Occlusal**
**Type 2 and type 1 patterns in adjacent areas (bar = 10 μm)**

**Figure 4**
**40%, Cervical**
**Pitted enamel surface, type 4 pattern (bar = 10 μm)**

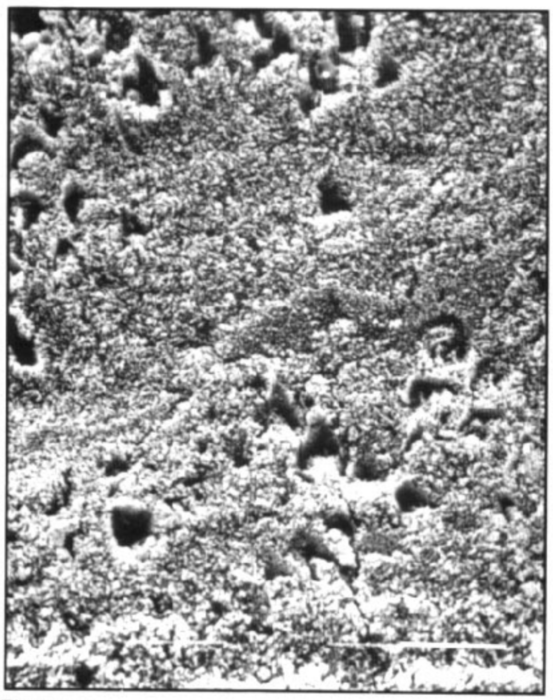

Figure 5

Figure 6

**Figure 5**
**40%, Cervical**
**Fine granulation of the enamel surface with numerous holes, type 5 and 4 patterns (bar = 10 μm)**

**Figure 6**
**20%, Central**
**Variation of etching patterns associated with the presence of perikymata (bar = 40 μm)**

## Results

### 40% solution

***Occlusal and central sites:*** The type 2 etching pattern with preferential dissolution of the prism peripheries predominates resulting in a "cobblestone" appearance; this is not always as clear and distinct as in Figure 1, but often more nonuniform and flat (Figure 2) or changing into type 1 (Figure 3). There are also isolated areas without prism structures showing only a fine roughening of the enamel surface (corresponding to type 3). Sometimes a variation of the etching patterns combined with the presence of perikymata can be seen.

***Cervical sites:*** The etching patterns are far more heterogeneous than in the occlusal and central third of the teeth. To a small extent, prism structures of type 2 can be observed (similar to Figure 2), but mainly pitted surfaces and map-like formations (type 4; Figure 4) or only a

**Figure 7**
**10%, Central 'Honeycomb' appearance, type 1 pattern (bar = 10 μm)**

**Figure 8**
**10%, Cervical Map-like formation, type 4 pattern (bar = 10 μm)**

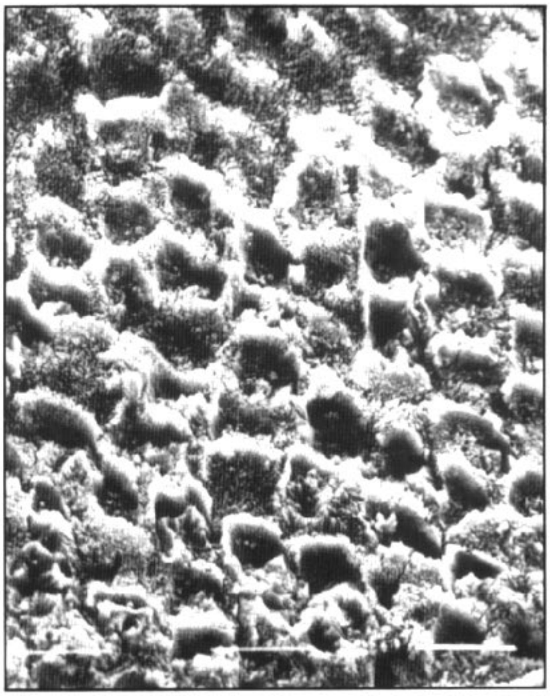

Figure 7

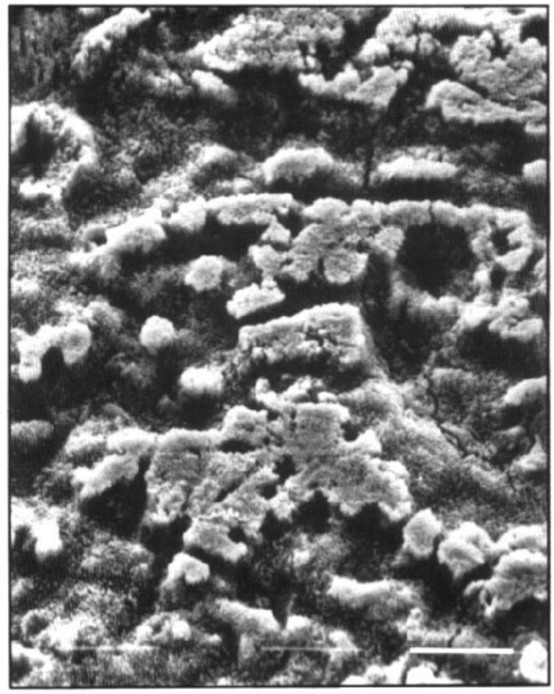

Figure 8

roughening of the crystallites appear (type 5; Figure 5).

**20% solution**

***Occlusal and central sites:*** The etching patterns are similar to those at 40% and range from very distinct prism delineation (mainly type 2) to aprismatic surfaces (types 4 and 5). The presence of perikymata is even more evident than at 40%. There are good etching effects on the crests and worse effects in the grooves (Figure 6; concerning the use of the terms perikymata and imbrication lines see Risnes[14]).

***Cervical sites:*** The morphology is nonuniform and similar to that at 40%. Mainly type 4 and 5 patterns occur, prism outlines are only rarely observed.

**10% solution**

***Occlusal and central sites:*** The etching patterns are again very heterogeneous and range from good prism delineation (type 1 more common than type 2; Figure 7) to aprismatic surfaces (types 4 and 5).

***Cervical sites:*** The morphology is nonuniform. Partly map-like formations appear (Figure 8), partly type 5 patterns (Figure 9), and partly disordered prism structures.

**5% solution**

***Occlusal and central sites:*** The enamel appearance is similar to that at 10% and contains type 1, 4, and 5 formations (Figure 10). But the "honeycomb" patterns of type 1 are less distinct than at 10%.

***Cervical sites:*** Only type 4 and 5 etching patterns are observed.

**2% solution**

***Occlusal and central sites:*** Also at this low concentration, light prism structures of type 1 become evident (Figure 11), additionally, aprismatic surfaces of types 4 and 5.

***Cervical sites:*** Predominantly type 5 etching patterns (Figure 12). At higher magnification the roughening of the crystallites is clearly distinguishable (Figure 13). Type 4 formations are only rare.

## Discussion

As reported in many previous studies, the present investigation has demonstrated great variation of the morphology of etched enamel surfaces. Using the same etching procedures, the etching patterns changed considerably from one tooth to another and also between directly adjacent areas of the same tooth. For this reason it appeared to be impossible to quantitate the extent of surface irregularity as done for example by Brännström et al.[15] This valuation is in agreement with Denys and Retief.[16]

The etching patterns were generally similar in the occlusal and central part of the buccal surfaces and frequently differed from the cervical area. Galil and Wright[13] reported that type 4 and 5 patterns (corresponding to type 3 of Silverstone et al.[12]) predominated in the cervical regions and types 1, 2, and 3 in the occlusal and central regions. Denys and Retief[16] made equiva-

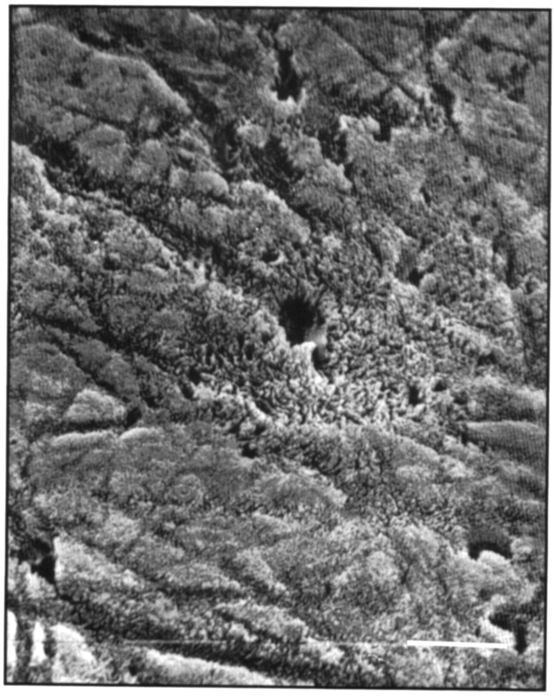

Figure 9

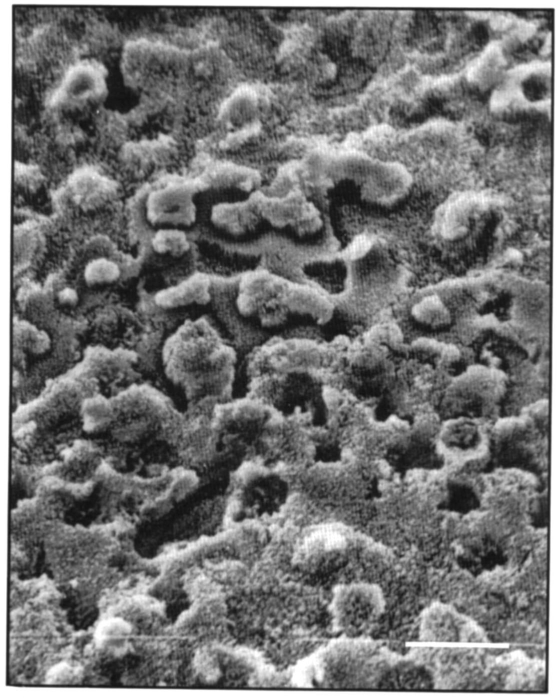

Figure 10

**Figure 9**
**10%, Cervical**
**Aprismatic etching with surface roughening, type 5 pattern (bar = 10 μm)**

**Figure 10**
**5%, Central**
**Aprismatic etching, type 4 pattern (bar = 10 μm)**

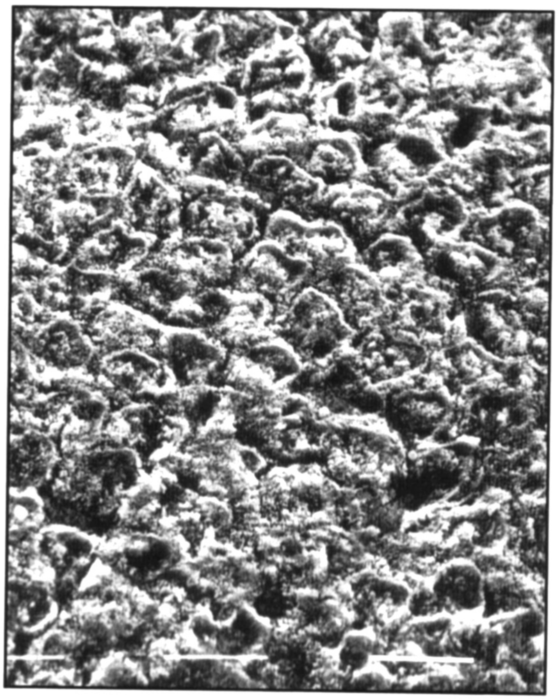

Figure 11

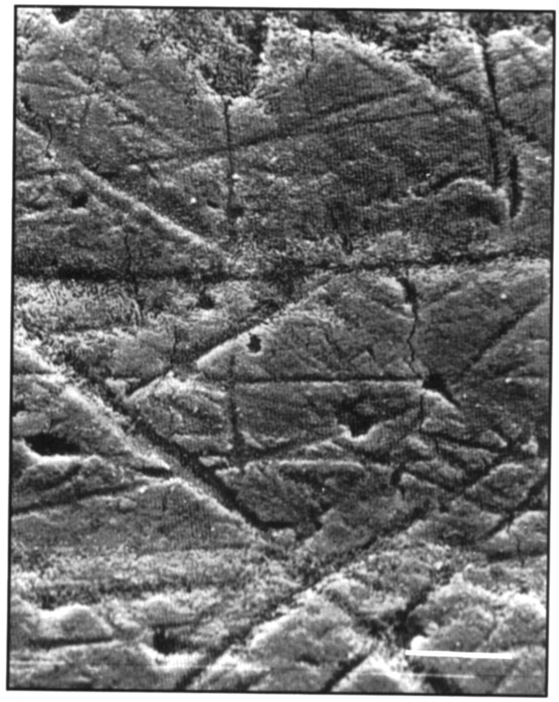

Figure 12

**Figure 11**
**2%, Occlusal**
**Superficially outlined type 1 pattern (bar = 10 μm)**

**Figure 12**
**2%, Cervical**
**Aprismatic etching with fine surface roughening, type 5 pattern (bar = 10 μm)**

lent findings. This is confirmed also by the present study. The type 4 and 5 patterns show no prism delineation and occur normally in areas of the so-called "prismless" enamel.[17] Ripa et al.[18] detected "prismless" enamel on 70% of the permanent teeth examined, primarily in the cervical third. The aprismatic layer is of variable thickness and appears in the microscope as a homogeneous, or more commonly, as a laminated band. The crystallites within the layer are oriented with their c-axis almost perpendicular to the enamel surface.[17-20] The increased occurrence of aprismatic enamel in the cervical region is probably also a consequence of reduced attrition in this area. Arakawa et al.[21] reported that the resin penetration was considerably worse in the cervical region than in the central and occlusal regions.

Galil and Wright[13] characterized the type 5 etching pattern as a flat, smooth surface with-

**Figure 13**
**2%, Cervical**
**Higher magnification of Figure 12. Surface roughening can be clearly distinguished (bar = 2 μm)**

Figure 13

**Table 1**
Observed types of etching patterns at different phosphoric acid concentrations in the occlusal, central, and cervical region (listed in order of frequency of occurrence).

| | occlusal, central | cervical |
|---|---|---|
| 40% | 2 > 1 > 3 > 4 > 5 | 4 > 5 > 2 |
| 20% | 2 > 1 > 3 > 4 > 5 | 4 ~ 5 > 2 |
| 10% | 1 > 2 ~ 3 ~ 4 ~ 5 | 4 ~ 5 > 1 ~ 2 |
| 5% | 1 ~ 4 ~ 5 | 4 ~ 5 |
| 2% | 1 ~ 4 ~ 5 | 5 > 4 |

out microirregularities. But normally a fine roughening on the crystallite level with microporosities can be observed (Figure 13). The sequence of etching patterns obtained with different acid concentrations gave the impression that the type 2 pattern, i.e. increased dissolution of the prism peripheries, occurred predominantly at higher acid concentrations (40% and 20%), whereas the softer etching procedures effected mainly the type 1 pattern with preferential dissolution of the prism cores. In accordance with that, Diedrich[22] supposed that the type 2 pattern seemed to represent an advanced stage of the etching process possibly caused by a breakdown of the fragile prism peripheries. Similar two-stage models have also been discussed by other authors.[23,24] It is reported in the literature[25,26] that the etching pattern depends primarily on the crystal orientation. The crystals arranged parallel to the attack were found to be the most vulnerable due to a weaker core along the entire c-axis. Therefore, an acid attack proceeding parallel to the direction of the prism lines produces a preferential dissolution of the cores leaving the "borders" or "tails" comparatively intact. This results in a "honeycomb" topography. It is imaginable that, if the etching process goes on, the acid attack will now also proceed perpendicular to the direction of the prism lines effecting a breakdown of the "honeycomb" structures.

The tensile bond strength measurements of Soetopo et al.[5] and Zidan and Hill[6] showed no significant differences between the 60-second etching with 35-40% and 2% phosphoric acid solutions. Unfortunately, these in-vitro tests cannot be compared directly with the clinical situation, where the bonded attachments are submitted to forces from various directions. Furthermore, bond strength measurements are usually carried out on ground, flat enamel surfaces, which will produce values different from those of unground teeth.[27,28]

A review of the literature concerning the relationship between the type of etching pattern and bond strength seems to indicate that regular and distinct type 1 and type 2 patterns provide maximum adhesion. Denys and Retief[16] however stated that it is not possible to define an etched enamel surface as retentive to dental resins only on the distribution of the etching patterns. The present investigation seems to confirm this observation.

Chow and Brown[29] reported that phosphoric acid concentrations greater than ~27% produce monocalcium phosphate monohydrate as a soluble product, while concentrations less than ~27% form dicalcium phosphate dihydrate. The former product is readily soluble and would be completely washed away in the clinical situation, whereas the latter product is less soluble. That seems to be a disadvantage to low acid concentrations. On the other hand the total loss of superficial enamel, which is especially rich in fluoride,[30] is probably smaller after etching with 2% or 5% phosphoric acid than with a 40% solution.[6,24,28,31] Also, the depth of acid penetration into deeper enamel layers seems to be smaller at low acid concentrations.[5] This could diminish the risk of decalcification in the enamel regions around the attachments.

Further studies are necessary to enlighten these subjects.

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