# Etching Treatment Effect on Surface Morphology of Dental Structures

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*This study examined and compared surface of human dentine after acidic etching with hydrogen peroxide, phosphoric acid liquid and gel. Surface demineralization of dentin is necessary for a strong bond of adhesive at dental surface. Split human teeth were used. After application of mentioned substances at dentin level measures of the contact angle and surface morphology were employed. Surface morphology was analyzed with the help of scanning electron microscopy and atomic force microscopy. Liquid phosphoric acid yielded highest demineralization showing better hydrophobicity than the rest, thus having more contact surface. Surface roughness are less evident and formed surface micropores of 4 μm remained open after wash and air dry providing better adhesive canalicular penetration and subsequent bond.*

*Keywords: dentin demineralization; dentin etching; phosphoric acid for dentin etching*

Durability and efficiency of dental indirect restorations is determinated by a strong bond of resin to repaired dental structures [1]. Dentin-resin bond is more susceptible to fatigue and failures then bond to enamel or compared to resin-ceramic bond [2]. Dentine-resin bond results from infiltration and polymerization of a synthetic resin at the level of dentine collagen fibers. This interaction forms a hybrid layer at the interface dentin-resin.

Dentin forms the bulk of the tooth structure, it is protected at surface by a layer of enamel, which is the hardest compound in the human body. Dentin protects dental pulp, the soft core of the teeth which is composed of nerves, vessels, mesenchymal cells and fibroblasts [3]. Dentin is less mineralized than enamel thus less hard but more than bones or cement which surrounds teeth root. Dentin is composed from two phases: inorganic one made of hydroxyapatite crystals and organic mostly represented by collagen. Dentin microstructure is made by a high number of tubules and channels separated by a calcified matrix [4,5].

At the dentin-resin interface a hybrid layer is formed. Hybrid layer will seal dentin surface and prevent secondary cavities, post intervention pain and it action as an elastic interface compensating tensions generated by contraction of resin polymerisation [6].

Dentine resin permeability has a crucial importance for a strong resin bond. In order to make dentin permeable and hydrophilic mineral component has to be dissolved with acid or different chelators and washed away.

Channels for resin infiltration are created around collagen fibers by hydroxyapatite acidic removal. Resin penetration into tubules will seal them and bonding to their walls will augment resin strength.

Efforts are made in order to produce monocomponent resin. The sensitive step for monocomponent resin is demineralization process that is crucial for an increase bond. For an increase resin penetration dentin is treated first with an acid compound followed by washing, dry and resin application. Acid compound is included in monocomponent resins in order to simplify the process of bonding. Washing and dry step is not needed for this type of resins, application process is faster but their ability to demineralized dentin is decreased [1]. Increased viscosity of these resins result in superficial interaction and incomplete demineralization thus limited permeability for resin monomers in dentin channels and tubules [8,9].

The aim of this study is to investigate etching treatments effects on the surface properties of dentin in order to evaluate bonding strength of different resins. We have used three etching substances to demineralize dentin: hydrogen peroxide, phosphoric acid gel and phosphoric acid liquid.

## Experimenral part

*Materials and methods*

We have used three etching compounds on dentine: hydrogen peroxide, phosphoric acid liquid 37% and phosphoric acid gel 36%. These substances were tested on sectioned human teeth, mono and pluri radicular. All teeth used were less than six months post extraction. All teeth were sectioned on mesiodistal direction using a water cooled electric saw. All the samples investigated were given a code number which can be found in table 1.

**Table 1**
CODE NUMBERS OF INVESTIGATED SAMPLES

| Code number | Etching type | Dental area tested |
|---|---|---|
| $D_1$ | Hydrogen peroxide | Dentine |
| $D_2$ | Phosphoric acid gel | Dentine |
| $D_3$ | Phosphoric acid liquid | Dentin |

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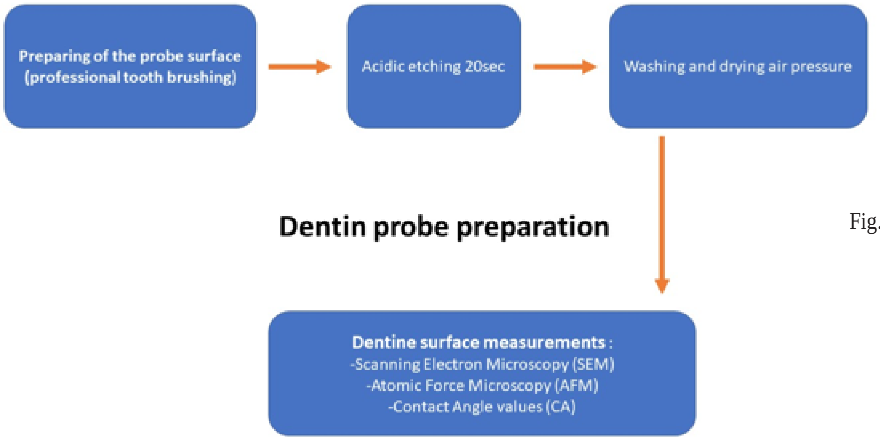


Fig.1. Overview of sample preparation and measurement

To investigate the effectiveness of dentine demineralization and morphological surface characteristics after acidic etching there have been carried out a series of investigations, to investigate the morphology of the surface has been used Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) while umectability properties were determined using Contact Angle values (CA) [10].

*Acidic etching*

A very important step for the study was the preparation of samples for investigation. For this step were selected 3 teeth mono and multi-radicular which were sectioned for the samples to be produced. It has been pursued reproduction of the process in dental praxis, using a process as close as possible to the real one that provide information about the effectiveness of the dentin etching in dental praxis and not under laboratory ex-vivo conditions [11].

Restoration process in dental praxis is composed of different steps:

- professional dental brush;
- isolation with dental dam and suction to maintain a dry working area;
- etching with hydrogen peroxide and phosphoric acid gel and liquid 35-37%;
- washing of the surface with water under pressure and drying with pressurized air;
- after acidic etching dentinal surface should have a chalk appearance;
- etched dentinal surface has to stay dry in order to apply bonding resin in the next step of restoration.

Demineralized dentin should stay dry and contamination of any kind should be avoided. If the chalky aspect is not seen or contamination occurs then the etching process has to be repeated. The bonding resin applied on the demineralized dentin surface will infiltrate channels and tubules forming the hybrid interface [7].

The overview of the process of sample preparation and measurement is presented in figure 1.

## Results and discussions

*Contact angle values*

In order to determine hydrophilic/hydrophobic characteristics, contact angle values were assessed using a Drop Shape Analyzer DSA 30 (Kruss GmbH, Hamburg Germany). Determination of surface free energy using Fowke's method generated around 200 values in 10 s time for each of three etching solutions table 2 [12,13].

Results shows that etching with hydrogen peroxide, D1 probe, and with phosphoric acid gel D2 demineralized dentin surface shows hydrophobicity. In case of probe D3 contact angle, value was 43.4°within the hydrophilic range.

D1 and D2 probes which represents reaction with hydrogen peroxide and phosphoric acid gel exhibit a low surface energy and a high contact angle. Probe D3 liquid phosphoric acid exhibit high surface energy and low contact angle thus favoring umectability.

*Scanning electron microscopy (SEM) measurements*

For probe surface analyze we have used scanning electron microscopy Quanta Inspect F (FEI-Thermo Fisher Scientific, California USA). Working parameters were 30Kv and 0.7 Torr pressure, all surfaces were gold smear covered [14]. Images can be seen in figures 2,3.

**Table 2**
HYDROPHILIC/HYDROPHOBIC CHARACTERISTICS

| Sample | Contact angle | | | Surface free energy | | |
|---|---|---|---|---|---|---|
| | Deionized water | Diiodometan | Ethilen glicol | $\gamma_s^{tot}$ [m N/m] | $\gamma_s^{p}$ [m N/m] | $\gamma_s^{d}$ [m N/m] |
| D1 | 81.0 | 45.5 | 65.0 | 31.4 | 12.2 | 19.2 |
| D2 | 80.8 | 70.3 | 81.8 | 27.2 | 16.8 | 10.4 |
| D3 | 43.4 | 52.4 | 52.4 | 55.4 | 35.4 | 20.4 |

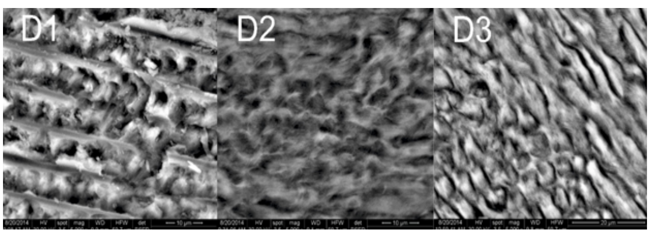


Fig. 2. SEM 5000x zoom: $D_1$, $D_2$, $D_3$

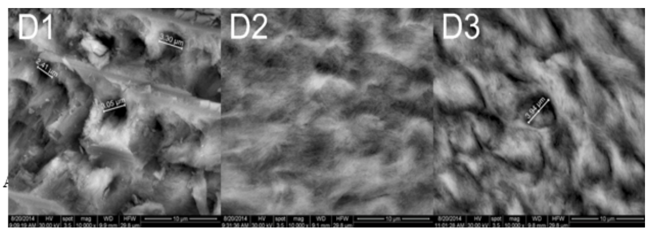


Fig. 3.SEM 10000x zoom: $D_1$, $D_2$, $D_3$

| Probe | Ra[nm] | Rq[nm] |
|---|---|---|
| $D_1$ | 16.6 | 19.6 |
| $D_2$ | 19.2 | 23.4 |
| $D_3$ | 12.5 | 16.4 |

**Table 3**
SURFACE ROUGHNESS VALUES WITH AFM MICROSCOPY

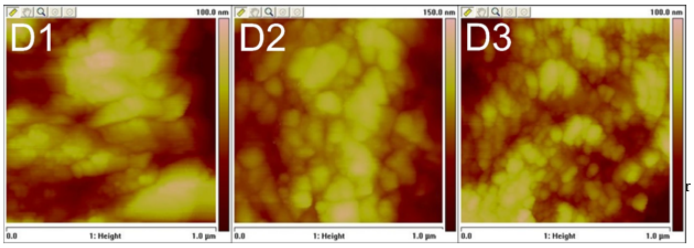


Fig. 4. 2D images from AFM microscopy: $D_1$, $D_2$, $D_3$

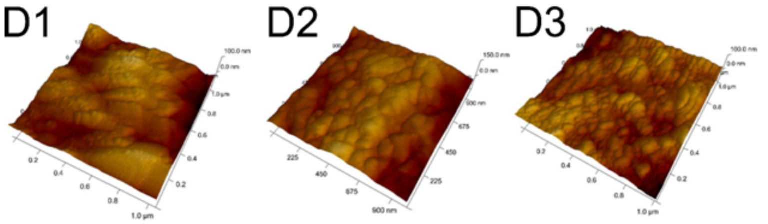


Fig. 5. 3D topography images from AFM microscopy: $D_1$, $D_2$, $D_3$

In order to analyze surface topography and to determined surface roughness we have used Atomic Force microscopy Veeco Multi Mode VS-AM (Vecco Instruments Inc., NY USA) [15]. Surface roughness values on table 3.

Surface roughness values are in line with results derived from scanning electron microscopy images. Etching with hydrogen peroxide and phosphoric acid gel determines high surface roughness values because of insufficient dentin penetration. For the liquid phosphoric acid lower values had been recorded because of better penetration in channels and tubules with consecutive demineralization [16].

With AFM microscopy, we have obtained 3D topography images of the probes surface after etching. .

Images obtained using scanning electron microscopy shows the effect of acidic etching on dentine using three solutions. It can be seen that etching with hydrogen peroxide (D1) weakens dentine structure, from water jet washing and drying under pressure some offices are destroyed. In the case of etching with phosphoric acid gel (D2) demineralization is not sufficient, because of high viscosity the acid does not penetrates into dentine channels and tubules producing a superficial demineralization thus does not create a favorable substrate for application of dental adhesive [17]. These results are consistent with the high contact angle and low surface free energy values, which shows hydrophobicity character of hydrogen peroxide and phosphoric acid gel. The best results were obtained in the case of etching with liquid phosphoric acid. Etching led to formation of holes of about 4 µm, which survived intact after washing with water and drying with air jet, so the adhesive can penetrate into demineralized dentine channels and tubules. Demineralized dentine surface is hydrophilic with low values of contact angle. Low viscosity of the liquid phosphoric acid determines an increased contact surface with dentin [18].

## Conclusions

All three investigated substances determine demineralization of dentine but the liquid phase of phosphoric acid produce the best results [19].

The use of different techniques of investigating dentine surface after etching allowed effect quantification of the three substances investigated [2]. Results showed hydrogen peroxide minimal penetration and low dentine demineralization. Phosphoric acid gel type produces a partial demineralization on dentine surface but because of high viscosity cannot penetrate deep [21]. Liquid phosphoric acid type yields the best results creating a hydrophilic dentine surface after demineralization thus providing best of all three surface for resin bond.

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