Comparative evaluation of Tannic Acid, Citric Acid and Phosphoric Acid as Etching agents for direct bonding

D. N. Kapoor* / V. P. Sharma** / Pradeep Tandon*** / Kamlesh Pandey****

The present study was undertaken to investigate the effect on enamel surface produced by the three etching agents i.e. 37% phosphoric acid, 50% citric acid and 50% tannic acid which was introduced for the first time in clinical orthodontics. One hundred and twenty five teeth (sixty one premolars and sixty four incisors) were used to study the efficacy of various etching agents on the enamel surface, assess the penetration depth and tensile bond strength.

It was determined from the present study that application of 37% phosphoric acid for 15 seconds produced comparable etching topography with that of 50% tannic acid when applied for 90 seconds. Electrothermal debracketing (ETD) which was carried out in the present study held advantage over conventional bracket debonding pliers. ETD produced bond failure at adhesive bracket interface with no iatrogenic damage to enamel. 50% Tannic acid provided tensile bond strength closer to 37% phosphoric acid and it was ascertained that it could be an alternative to conventional phosphoric acid etching as it dissolved lesser enamel and provided similar bond strength values.

Introduction:

With the advent and application of direct bonding techniques, a new era dawned as it opened new horizons in the specialty of Orthodontics both in the clinical and research field. The future of bonding is promising as new avenues are opening up but there is still remarkable lack of consensus regarding clinical bond strength values and the effects of conventional acid etching. The technique utilises a wide variety of acrylic or composite resins to bond the brackets directly to the etched enamel surface and maintain them firmly throughout the duration of the treatment. Initially the technique was plagued by frequent bond failures and a constant effort to improve the bond strength was envisaged. Inspite of the introduction of high filled diacrylate resins which impart greater bonding strength, a high rate of attachment failure continued to be a common feature. Various studies have established that the etching of enamel is more important than the strength of resins to achieve a reliable bond.

The acid etch technique has undergone a vast change since being introduced by Buonocore (1955)1. The efficacy of a number of organic and inorganic acids along with chelating agents of different concentrations and the duration of their application have been evaluated in an effort to obtain optimal etching of the enamel surface.

The present study approaches the problem by investigating the effect on enamel surface produced by the three etching agents i.e. 37% phosphoric acid, 50% citric acid and 50% tannic acid.

The objectives of the present study were:

1. To determine the effect on enamel surface after etching, debonding and clean up through scanning electron microscope.
2. To assess the penetration depth.
3. To assess the tensile bond strength.

Material and Method:

The sample comprised of one hundred and twenty five freshly extracted healthy teeth (sixty one premolars and sixty four incisors). They were used to study the efficacy of various etching agents on enamel surface, for direct bonding of orthodontics attachments.

* Professor and Head
** Professor
*** Associate Professor
**** Former Postgraduate Student

Department of Orthodontics, Faculty of Dental Sciences, KG's Medical College, Lucknow.
The criteria for selection of the teeth was that they be vital and fully erupted, free from caries and restorations.

Material:

The etching solutions used throughout the study were prepared from analytical grade reagent at Central Drug Research Institute, Lucknow and were coded as solution A, B and C.

(A) 37% Orthophosphoric Acid W/W
(B) 50% Citric Acid W/W
(C) 50% Tannic Acid W/W

Orthodontic stainless steel edgewise brackets having standard bracket slot .022"x.028" (R.M.O. U.S.A.) were welded on the fine gauze mesh (Dentaurum, W. Germany) with five spot welds, one at the center of the bracket and four on the corners. Distribution of samples of 125 extracted teeth is depicted in Table 1.

Material:

The etching solutions used throughout the study were prepared from analytical grade reagent at Central Drug Research Institute, Lucknow and were coded as solution A, B and C.

(A) 37% Orthophosphoric Acid W/W
(B) 50% Citric Acid W/W
(C) 50% Tannic Acid W/W

Orthodontic stainless steel edgewise brackets having standard bracket slot .022"x.028" (R.M.O. U.S.A.) were welded on the fine gauze mesh (Dentaurum, W. Germany) with five spot welds, one at the center of the bracket and four on the corners. Distribution of samples of 125 extracted teeth is depicted in Table 1.

Method:

Considering aims and objectives, the study was taken up into three parts:

(i) Effect on enamel surface — A Scanning Electron microscopic study.
(ii) Assessment of penetration depth.
(iii) Assessment of tensile bond strength.

(i) Effect on Enamel Surface — By Scanning Electron Microscope

Sixty one premolar teeth (maxillary and mandibular) were taken as samples. Distribution of samples for scanning electron microscopic study of effects on enamel surface after etching on varying duration is depicted in Table 2.

Etching:

Buccal surface of the samples were polished with pumice paste and rubber cup for ten seconds prior to etching. Samples were etched with the solution A, B and C for given time (Table no. 2). Etching time for every solution varied in order to compare the etching time and the quality of etch.

Etchant was applied with the help of a sponge pellet, thereafter it was washed off with 5 ml of distilled water and was then dried with hot compressed air and kept in a dessicator over night and then subjected to scanning electron microscopy.

Debonding:

Brackets were bonded to enamel surface of the sample group and left overnight for polymerization. Samples were subjected to Electro Thermal Debracketing (ETD), a controlled heat producing device, developed in the department. A temperature of 133°C was applied for 8 seconds. As the bracket resin interface deformed due to softening of resin, the bracket was gently lifted with the help of a bracket holding tweezer. Buccal surface of half the samples (Table no. 1) were carefully cut out longitudinally kept in dessicator overnight and then subjected to scanning electron microscopy.

Clean up:

Samples were subjected to clean up procedure. Gross adhesive was removed with the help of a tungsten carbide bur and finally polished with rubber cup and pumice paste for fifteen seconds.

After clean up, the buccal surface was carefully cut out longitudinally and kept in a dessicator overnight and then subjected to scanning electron microscopy.

Procedure:

All the sixty one specimens were observed under scanning electron microscope (Philips) one by one and scanned as a whole initially. The desired area was then focused and scanned at a magnification of SEM x 93 to SEM x 4020. The current used during scanning was 25 KV under a vacuum pressure of 1 x 10^-3 torr and representative photomicrographs were then obtained.

(ii) Assessment of Penetration Depth:

The depth of etching on the polished enamel surface produced by the three etching solutions was determined by surface profile recordings. The labial enamel surfaces of four maxillary incisors were polished with 600 grit silicon carbide paper washed thoroughly with distilled water and then dried with hot compressed air. The three incisors were etched with the solution A, B and C for 15 seconds, 60 seconds and 90 seconds respectively on the labial enamel surfaces. The surface profiles of these specimens were recorded with the help of a computerized Taylor- Hobson Talysurf model-6 surface roughness testing machine (Rank Precision Industries, England). A computer provided the mean maximum peak to valley height (Ry) in five sampling lengths and each sampling length was 0.25 mm.

(iii) Assessment of Tensile Bond Strength:

A square 2x2 cm stainless steel wire fixture 0.022"x 0.028" was made to hold the bonded bracket with the help of a 0.010" non prestretched ligature wire for the purpose of the tensile bond strength test.

The exposed tooth surface was etched for a period of 15 seconds for solution A, 60 seconds for solution B and 90 seconds for solution C. Meshed stainless steel edgewise brackets were bonded onto the etched enamel surface with Rely-a-bond adhesive material.
Instron Model TT-C (Instron Eng. Corp. Canton Mass) was allowed to warm up for 30 minutes prior to testing. The specimen was then checked for alignment and a load was applied with a cross head speed of 0.5 cm/minute. Care was taken to eliminate all forces except tensile load.

Results:
The findings are presented under the following headings:

1. Effects on Enamel Surface by Scanning Electron Microscope

Etching
Normal enamel surface showed featureless appearance when viewed in the scanning electron microscope (Fig. 1) at magnification x 93. Etching with 37% phosphoric acid for fifteen seconds at low magnification of x 93 revealed a characteristic regular streaky pattern due to greater etching of ridges than troughs in the regions of perikymata (Fig. 2). The same at higher magnification of x 1050 showed variable etching pattern (Fig. 3). In some areas the characteristic prism-end structure was observed which resulted from the preferential etching of prism cores, which provided a typical honeycomb appearance to the etched surface. Some photomicrographs revealed prism peripheries etched to a greater extent than prism cores (Fig. 4). Another constant feature observed on all enamel surfaces etched with 37% phosphoric acid was the presence of areas which showed no characteristic etching topography. In these zones of prismless enamel, prism patterns were not delineated (Fig. 5). Etching for more than fifteen seconds resulted in loss of superficial enamel texture and formation of clefts, pits and an ill defined etching pattern (Fig. 6).

Every etching pattern was obtained by etching the enamel surfaces with 50% citric acid for one minute (Fig. 7). At a higher magnification, the honeycomb appearance of the etched enamel was clearly demonstrated (Fig. 8). It was observed that citric acid preferentially etched the prism cores to a greater extent than the peripheries. Ninety second application of citric acid resulted in an atypical etch pattern which gave the appearance of bizarre fissures and grooves which is rarely seen (Fig. 9).

Etching with 50% Tannic acid produced a fairly even etching pattern of the whole surface (Fig. 10). The preferential etching of the tannic acid was mainly confined to prism cores resulting in the characteristic prism-end structure (Fig. 10). At a higher magnification, the typical honeycomb appearance of the etched surface was demonstrated (Fig. 11).

Scanning electron microscopic studies of enamel surface effects produced by various etching agents were alone not sufficient to derive any definite information regarding optimal etching time. Hence further assessment covering the full extent of interlocking between adhesive and etched enamel of penetration depth and assessment of tensile bond strength was carried out to arrive at a conclusion regarding the optimal etching time and concentration.

Debonding:
Samples were debonded with electrothermal debracketing technique and then were subjected to SEM investigations. Debonding procedure provided similar results irrespective of the etching solution used. All samples failed at adhesive bracket interface and no enamel damage was noticed (Fig. 12).

Clean up:
Adhesive left on enamel surface was removed with a tungsten carbide bur and final polishing was carried out with pumice paste and a rubber cup. As removal of adhesive was completed, surface scratches along with few adhesive remnants and occasional areas of etched enamel were visible (Fig. 13). Final polishing with pumice paste and rubber cup resulted in a texture of enamel with fine scratches with very few adhesive remnants left on the enamel surface (Fig. 14).

Thus scanning electron microscopy reveals electrothermal debracketing technique holding advantage over conventional bracket removing pliers as the site of bond failure was observed at the bracket adhesive interface. With this technique no iatrogenic damage to enamel and no bracket distortion was observed. Remaining adhesive left on enamel surface was successfully removed by a tungsten carbide bur operated at low speed in a dry field. Final polishing of the enamel surface with pumice paste and rubber cup resulted in smooth enamel surface (Fig. 14).

2) Assessment of Penetration Depth:
Surface profile measurements of unetched polished tooth and the teeth etched with solution A, B and C for 15 seconds, 60 seconds and 90 seconds respectively were carried out. Deepest penetration depth of 18.8 mm was obtained with 37% phosphoric acid and the least penetration depth of 5.7 mm was obtained with 50% citric acid, while measured depth of etch of 16.4 mm was obtained by 90 second application of 50% tannic acid (Table 3).

3) Assessment of Tensile Bond Strengths:
The sixty samples were divided into three groups i.e. group A, B and C treated with phosphoric acid, citric acid, tannic acid respectively (Table No. 1). Thereafter meshed stainless steel edgewise orthodontic brackets having a surface area of
Fig. 1: Polished unetched enamel at magnification (SEMx93).

Fig. 2: Enamel surface appearance after 15 second etching with 37% orthophosphoric acid. Magnification (SEMx93).

Fig. 3: Enamel surface etched with 37% phosphoric acid for 15 seconds at magnification SEMx1050.

Fig. 4: 15 seconds etching of enamel with 37% phosphoric acid showing central and peripheral etching patterns at magnification SEMx2300.

Fig. 5: Absence of typical etching pattern in areas of prismless enamel. 15 seconds etching with 37% phosphoric acid at magnification (SEMx1150).

Fig. 6: Etching with 37% phosphoric acid for 30 seconds (SEMx1050).

Fig. 7: Even etching pattern of enamel surface treated with 50% citric acid for 60 seconds (SEMx1150).

Fig. 8: One minute etching with 50% citric acid showing typical honeycomb appearance (SEMx4020).
Fig. 9: 50% citric acid application for 90 seconds (SEM x93)

Fig. 10: 50% Tannic acid application for 60 seconds (SEM x1150)

Fig. 11: 50% Tannic acid etching for ninety seconds showing typical honeycomb appearance (SEM x4020)

Fig. 12: Adhesive left on etched enamel surface at debonding (SEM x1150)

Fig. 13: Adhesive removal with tungsten carbide bur completed (SEM x1150)

Fig. 14: Clean up enamel surface (SEM x93)

7.5 mm² and 11 mm² were bonded on to etched enamel. Samples were subjected to tensile stresses and load required to break the bond was recorded in kilogram. Thereafter tensile bond strength was calculated.

Table 4 shows the tensile strength in megapascals (Mpa). The highest tensile bond strength values were 9.10 Mpa (± 1.95) obtained from Group A, whereas lowest was recorded for Group B, 5.82 Mpa (± 1.39). The tensile bond strength value of group C obtained was 9.09 Mpa, which was almost equal to the bond strength value obtained from group A.

The 't' test value was found to be insignificant in Group A versus Group C indicating thereby that application of 37% phosphoric acid for fifteen seconds on enamel surfaces provided almost equal tensile bond strength to that of 50% Tannic acid application for 90 seconds. Differences between group A versus B and Group B versus C were highly significant indicating that etching with citric acid had remarkable
low tensile bond strength in comparison to phosphoric acid or tannic acid etching. Table 5 shows statistical comparison of tensile bond strengths of Group A, B and C.

Discussion:

Dental enamel lacks in regenerative power and calls for great care in its maintenance. Buonocore (1955)\(^1\) etched the enamel surfaces with 85% phosphoric acid in order to obtain satisfactory mechanical bonding between resin and the etched enamel surface. Newman (1968)\(^2\) applied acid etch technique in orthodontics for direct bonding of orthodontic attachments. Results had been encouraging and since then significant advancements in adhesive technology have brought about a revolution. Brown (1978)\(^3\) and Pos (1980)\(^4\) indicated that orthodontic bonding procedure (etching, debonding and clean up procedure), produce approximately 55 mm loss of enamel. Zachrisson and Artun (1979)\(^5\) demonstrated that orthodontic bonding procedure resulted in aging of the enamel by 15 years. Smith (1986)\(^6\) emphasized over the use of organic acids as etching agents because these organic acids act at a slower rate with lesser penetration.

It is evident that acid etching of enamel has not yet been properly understood. Unfortunately no quantitative measures have been so far established to achieve optimal etching (Bright and Shannon, 1980)\(^7\).

In the present study phosphoric acid 37% and citric acid 50% were selected for the purpose of direct bonding. Further, tannic acid 50% has been tried for the first time in clinical orthodontics, though same has been used by Powis et al. (1982)\(^8\) in restorative dentistry. These etching agents were considered suitable for trial to avoid unnecessary loss of enamel. Etching time ranged from 15 to 60 seconds for phosphoric acid and 60 to 90 seconds for Tannic acid and citric acid.

Effects on Enamel Surface- By Scanning Electron Microscope

Etching:

Acid etching of enamel surface removes low energy organic film from the tooth surface and preferentially etches the enamel surface creating interprismatic or intraprismatic spaces, thereby increasing the wettability of the enamel surface (Gwinnett, 1967; Buonocore, 1968; Retief, 1973\(^9\)). Etching with 37% phosphoric acid produced variable type of etching pattern. In some areas, prism cores were dissolved to a greater extent (Fig. 3) corresponding to pattern I or central etch type reported by Silverstone et al., (1975)\(^10\), while in some areas prism peripheries were etched preferably (Fig. 4) producing pattern II or peripheral etch type. Further in zones of prism less enamel no definite pattern was obtained. Only dissolution of superficial enamel was seen (Fig. 5), which has been classified by Silverstone (1975)\(^10\) as less structured type of etching pattern or pattern III. Results are similar to that obtained by (Retief, 1975)\(^11\), Retief et al. 1976\(^12\), Beech et al. 1980\(^13\), Brannstrom et al., 1982\(^14\), Barkemeir, 1985\(^15\). Diedrich (1981)\(^16\) reported additional etching pattern i.e. Type IV which he named star or fern like etching pattern, which in the present study was not observed.

Etching time of more than fifteen seconds resulted in formation of clefts, grooves, pits and ill defined etching pattern (Fig. 5, 6). Brannstrom et al., 1982\(^17\) however noticed no difference in appearance of enamel treated with 37% phosphoric acid from 15 to 120 seconds.

Beech and Jalay (1980)\(^18\) found five second application of 50% phosphoric acid provided optimal results. Diedrich (1981)\(^19\) however noticed loss of superficial enamel when etching time exceeded 30 seconds. It is certainly desirable to dissolve the minimal amount of enamel consistent with optimum bond strength.

Citric acid and Tannic acid produced a much more even etching pattern where prism cores were etched to a greater extent than prism peripheries (Fig. 7,8,10,11). H. Lee and Swartz (1971)\(^20\) compared 50% H\(_3\)PO\(_4\) and 50% citric acid by exposing the enamel surface to these etching solutions for varying duration and found that phosphoric acid dissolved the enamel at faster rate. This was subsequently confirmed quantitatively by Retief (1974)\(^21\). The perikymata demonstrated a variable etching pattern where ridges were etched to a greater extent than troughs (Fig. 2) which is in conformity to the findings reported by Retief (1974)\(^20\), Retief et al. (1976)\(^22\) and Diedrich (1981)\(^19\).

50% citric acid had the mildest etching action amongst the three conditioning solutions evaluated in this investigation. Etching time of fifteen seconds with 37% phosphoric acid and ninety seconds with 50% tannic acid resulted in comparable etching topography.

Debonding:

Debonding is the stage of the orthodontic bonding process which has evoked constant criticism and comments from practitioners. Debonding with highly filled resins is difficult, time consuming and damaging to the enamel (Gorelick, 1977) and it is an established fact that conventional bracket removing pliers are known to produce enamel
cracks and tears because of the peeling force applied on the adhesive enamel aspect (Zachrisson, 1980; Diederich, 1981; Sandison, 1981).

Electro thermal debracketing (ETD) was carried out in the present study, which transferred controlled heat to the bracket resin interface producing a temperature of 133°C for 8 seconds which is close to the softening temperature of filled composite adhesive materials i.e. 126°C and at the same time much below the pulpal heat threshold of primates i.e. 277°C. ETD holds advantage over conventional bracket removing pliers. These observations of the present study are in agreement with Sheridon et al. (1986). Further, effectiveness of electro thermal debracketing (ETD) on plastic and ceramic brackets is questionable as these are poor conductors of heat.

**Clean up**

It has been claimed by many researchers (Fitzpatrick and Way, 1977) that any of the adhesive left after debonding is abraded by normal wear. However, clinical experience does not support this claim. Zachrisson and Artun (1979) found no reduction in amount of adhesive material over periods of a year.

In the present study a clean up procedure was successfully completed with tungsten carbide bur operated at low speed in a dry field. The procedure was carried out as described by Sandison (1981) in a dry field.

Photo micrographs (Fig. 14) obtained after completion of clean up procedure (Fig. 15) are similar to those obtained by Retief et al., 1979 and Sandison, 1981. It seems probable that tags of resin remain on the enamel surface following clean up although the tooth appears clinically clean (Caspersen, 1977; Sandison, 1981).

**Assessment of Penetration Depth:**

Assessment of penetration depth was carried by measuring surface profile recordings of etched enamel with Tayler Hobson Talysurf Model-6 surface roughness testing machine. This technique has been previously used by Retief, 1974; Retief, 1975; and Retief et al., 1976. The diameter of stylus was not so fine to record the finest details. Thus only a comparative conclusion can be drawn i.e. 37% phosphoric acid penetrated the deepest and 50% citric acid penetrated the least.

Citic acid and Tannic acid penetrated to a depth of 5.7 μm and 16.4 μm respectively. However there is no experimental evidence to support this, as penetration depth of these acids were observed for the first time.

Penetration depth was 18.8 mm when 37% phosphoric acid was applied for 15 seconds, comparing favourably with that of Retief (1975) where the penetration was 21.7 mm with 50% phosphoric acid for 60 seconds. The greater concentration and longer application leads to the greater etching depth.

**Assessment of Tensile Bond Strength:**

A sub committee on standard test methods for Dental Materials Group of the IADR decided in the year 1967 that a tensile test should be used in preference to other tests. To control the effects of many variables, the tooth surfaces were flattened to determine the bond strength advocated by Mizrahi et al., 1971, as all the forces acting on an adhesive attached to the enamel can be resolved into components at right angles (Tensile) and parallel (shear) to the enamel surface.

Bond strengths of the two acids were evaluated in order to find out if any of the acids can be used with an efficacy comparable to that of phosphoric acid. The 15 second application of 37% phosphoric acid on enamel surface provided the mean tensile bond strength.

The results of this study indicate that either a fifteen second treatment with 37% phosphoric acid or a ninety second treatment with 50% tannic acid provide the adequate orthodontic tensile bond strength, at the same time, removing less amount of healthy enamel. Tannic acid was investigated for the first time in orthodontics and further studies would be better for its use in the clinical practice.

**Conclusion:**

Following conclusions were drawn from the present study:

1. Effects on enamel surface produced by three etchants revealed through scanning electron microscope.
   (a) Application of 37% phosphoric acid produced variable etching pattern.
   (b) Citric acid 50% produced central type of etching pattern.
   (c) Application of 50% Tannic acid produced central type of etching pattern.
   (d) Application of 37% phosphoric acid for 15 seconds produced comparable etching topography when 50% tannic acid was applied for 90 seconds.
   (e) Electrothermal debracketing technique produced bond failure at adhesive bracket interface with no iatrogenic damage to enamel.
   (f) Tungsten carbide bur at low speed in a dry field followed by pumice polishing was effective for removal of adhesive left on enamel surface.
2. Assessment of penetration depth revealed that 37% phosphoric acid dissolve more enamel than tannic acids or citric acid.
3. Assessment of tensile bond strength reveals that (a) Phosphoric acid 37% when applied on the enamel for 15 seconds provided adequate bond strength at adhesive enamel interface. (b) One minute application of 50% citric acid did not result in attainment of adequate bond strength. (c) Tannic acid 50% when applied on enamel for ninety seconds provided the tensile bond strength closer to 37% phosphoric acid.
4. Tannic acid 50% could be an alternative to phosphoric acid for etching because it dissolves lesser amount of enamel and at the same time provides similar tensile bond strength.

Table 1: Showing distribution of 125 samples of extracted teeth

<table>
<thead>
<tr>
<th>PART I</th>
<th>PART II</th>
<th>PART III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Effects of enamel surfaces—By scanning electron microscope (61 premolar teeth)</td>
<td>Assessment of penetration depth (4 incisor teeth)</td>
<td>Assessment of tensile bond strength (60 incisor teeth)</td>
</tr>
<tr>
<td>Etching</td>
<td>Debonding</td>
<td>Group</td>
</tr>
<tr>
<td>Control Group</td>
<td>1</td>
<td>A</td>
</tr>
<tr>
<td>Experimental Group</td>
<td></td>
<td>B</td>
</tr>
<tr>
<td></td>
<td></td>
<td>C</td>
</tr>
</tbody>
</table>

Table 2: Showing distribution of samples for scanning electron microscopic study of enamel surface effects

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Solution</th>
<th>Etching time (in Seconds)</th>
<th>No. of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Etching</td>
<td>A</td>
<td>15</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>60</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>60</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>60</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90</td>
<td>5</td>
</tr>
<tr>
<td>Debonding</td>
<td>A</td>
<td>15</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>60</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>90</td>
<td>10</td>
</tr>
<tr>
<td>Polished</td>
<td></td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>unetched</td>
<td></td>
<td></td>
<td>1</td>
</tr>
</tbody>
</table>

Table 3: Depth of etching measurements from surface profiles

<table>
<thead>
<tr>
<th>Etching Solution</th>
<th>Measured depth of etch in (µm) n=1</th>
<th>Etching time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unetched Polished enamel</td>
<td>4.8</td>
<td></td>
</tr>
<tr>
<td>A 37% H₃PO₄</td>
<td>18.8</td>
<td>15 sec.</td>
</tr>
<tr>
<td>B 50% Citric acid</td>
<td>5.7</td>
<td>60 sec.</td>
</tr>
<tr>
<td>C 50% Tannic acid</td>
<td>16.4</td>
<td>90 sec.</td>
</tr>
</tbody>
</table>

Table 4: Showing tensile bond strength in Mpa

| Group | n=1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | Mean | S.D. |
|-------|-----|---|---|---|---|---|---|---|---|----|----|----|----|----|----|----|----|----|----|-----|------|
| A     |     | 11.00 | 9.02 | 7.68 | 13.49 | 11.00 | 8.10 | 10.32 | 10.59 | 7.79 | 12.70 | 6.93 | 7.50 | 8.59 | 9.57 | 6.70 | 9.89 | 7.06 | 6.74 | 8.45 | 8.92 | 9.10 | 1.95 |
| B     |     | 6.87 | 7.11 | 4.73 | 5.07 | 7.34 | 4.91 | 7.34 | 7.84 | 4.94 | 5.46 | 5.13 | 3.44 | 3.56 | 5.93 | 7.53 | 3.81 | 6.27 | 6.75 | 7.14 | 5.33 | 5.82 | 1.39 |
Table 5: Showing statistical comparison of tensile bond strengths of Group A, B and C

<table>
<thead>
<tr>
<th>Group</th>
<th>A Mean</th>
<th>A S.D.</th>
<th>B Mean</th>
<th>B S.D.</th>
<th>C Mean</th>
<th>C S.D.</th>
<th>t value</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A versus Group B</td>
<td>9.10</td>
<td>1.95</td>
<td>5.82</td>
<td>1.39</td>
<td>-</td>
<td>-</td>
<td>6.12***</td>
<td></td>
</tr>
<tr>
<td>Group A versus Group C</td>
<td>9.10</td>
<td>1.95</td>
<td>-</td>
<td>-</td>
<td>9.09</td>
<td>1.93</td>
<td>0.01</td>
<td>NS</td>
</tr>
<tr>
<td>Group B versus Group C</td>
<td>-</td>
<td>-</td>
<td>5.82</td>
<td>1.39</td>
<td>9.09</td>
<td>1.93</td>
<td>6.11***</td>
<td></td>
</tr>
</tbody>
</table>

*** t value highly significant

References: