



## Effect of Trichloroacetic Acid Hydrogel on Self-Etch Adhesive Bond Strength to Dental Tissues

Kamyar Fathpour, Maryam Khoroushi

### ABSTRACT

**Introduction:** Trichloroacetic acid (TCA) is a soft tissue cauterizing agent applied to gingival margins prior to cervical tooth-colored restorations. The present *in vitro* study evaluated the effects of two different concentrations of TCA hydrogel as a hemostatic/preconditioning agent on the shear bond strength (SBS) of a self-etch adhesive to tooth structures.

**Materials and methods:** Thirty-six flat enamel and 36 flat dentin surfaces were prepared using human molars; each group was subdivided into three subgroups ( $n = 12$ ). The groups were made ready as follows: In groups 1 (E1 and D1), the enamel (E) and dentin (D) surfaces were designated as control groups and remained intact. In groups 2 (E2 and D2), 35% TCA gel was applied to enamel and dentin surfaces for 30 seconds. In groups 3 (E3 and D3), 50% TCA gel was applied to enamel and dentin surfaces for 30 seconds. Clearfil SE Bond and Z100 composite resin were applied to the surfaces according to manufacturers' instructions. After 24 hours of incubation and thermocycling, the composite cylinders underwent an SBS test in a DARTEC test machine. Data were analyzed using the ANOVA and Scheffé's test ( $\alpha = 0.05$ ).

**Results:** The mean SBS  $\pm$  SD in the study groups were  $34.73 \pm 5.66$ ,  $35.32 \pm 7.3$ ,  $23.75 \pm 9.67$ ,  $20.94 \pm 9.84$ ,  $19.32 \pm 6.20$ ,  $23.50 \pm 6.63$  MPa in the E1, E2, E3, D1, D2 and D3 groups, respectively. ANOVA revealed significant differences between the SBS values of enamel groups ( $p = 0.001$ ). There were no significant differences between the dentin groups ( $p = 0.425$ ).

**Conclusion:** Application of 35 and 50% concentrations of TCA to dentin had no detrimental effect on the bond produced by two-step self-etch adhesive under study; however, application of only 35% TCA to enamel did not result in a detrimental effect on the same adhesive.

**Keywords:** Acid preconditioning, Bond strength, Composite resin, Contamination, Hemostasis, Self-etch adhesive.

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**Conflict of interest:** None

### INTRODUCTION

Composite resins are extensively used in bonding procedures to tooth structures. They are commonly used in different oral cavity regions and various tooth surfaces. Demand for esthetic restorations on various tooth surfaces is ever increasing. On the other hand, with an increase in living standards and life expectancy all over the world, cervical carious lesions, including NCCL or root surface caries, are becoming more prevalent, necessitating esthetic cervical restorations.<sup>1</sup>

Bonding composite resin to tooth structures is a variable and technique-sensitive procedure. Detrimental effects of contaminants on bonding strengths further complicate the problem. Composite resin restorative procedures require an operating field free of any contaminants so that the highest possible bond strength would be achieved.<sup>1,2</sup> It has been reported that contamination with saliva, blood or crevicular fluids interferes with the wetting process and penetration of composite resin into the dentin surfaces conditioned by acid.<sup>3</sup>

In case of hemorrhage, a hemostatic agent is applied to accomplish a successful bonding procedure and achieve proper bond strength. In such clinical situations, hemostatic agents can control hemorrhage and gingival fluids.<sup>4</sup>

Traditionally, different techniques and materials have been used to control contamination with saliva and crevicular fluids. Epinephrine, aluminum chloride and ferrous sulfate are usually used to control hemorrhage and crevicular fluids. In addition, electrosurgical techniques and laser irradiation are used to this end. Each technique and material has disadvantages too.<sup>5,6</sup>

The pH values of these hemostatic agents are in the order of 0.7 to 3.0.<sup>4</sup> The tooth structure, in particular dentin as a major part of the preparation cavity, might be contaminated by these acidic hemostatic agents. As a result, some changes

might occur on enamel and dentin surfaces subsequent to contamination with these highly acidic agents, which include removal of the smear layer and demineralization of dentin and enamel.<sup>7-9</sup>

Trichloroacetic acid (TCA), as a hemostatic agent, is believed to provide efficient isolation from crevicular fluids, blood and saliva. TCA, a cauterizing chemical agent, has been effectively used in medicine and dentistry for a long time.<sup>10</sup> TCA is produced by chlorination of acetic acid. Its aqueous solution is highly acidic with a pH value of 1.0. It has been used as a caustic agent in medicine.<sup>10</sup> Highly saturated solutions of TCA have been used to control crevicular fluids and hemorrhage in the restoration of cervical area cavities.<sup>11,12</sup>

At present, two approaches are used in bonding procedures with tooth structures: Etch-and-rinse and self-etch techniques. Self-etching systems have incorporated an aqueous mixture of acidic functional monomers with acidity lower than those of phosphoric acid gels.<sup>13-15</sup> With the application of this bonding approach, residual smear layer and remnants of dissolved hydroxyapatite crystals can be seen within the hybridized complex.<sup>15</sup> These bonding systems have won more worldwide popularity among researchers and clinicians compared to the traditional etch-and-rinse systems because of simplified approach and less technique sensitivity.<sup>16,17</sup>

Several studies have shown the effect of conditioning substances prior to the application of self-etch adhesives.<sup>18-20</sup> Given the acidity of TCA, contamination of dental substrates with this material during control of hemorrhage in gingival tissues might have a detrimental effect on the bonding process of composite resins to cervical areas.

Recently, two studies evaluated the effect of TCA on bond strength of composite resin to enamel using an etch-and-rinse adhesive system. The results showed that TCA can etch enamel surfaces, resulting in favorable bond strength values with the use of Single Bond etch-and-rinse adhesive system.<sup>21,22</sup> In addition, contact of the material with enamel prior to the application of phosphoric acid might have a positive effect on immediate bond strength of composite resin to enamel with the use of etch-and-rinse adhesives.<sup>21</sup>

Since, self-etch adhesives are the materials of choice for cervical restorations and TCA might be used to control gingival hemorrhage, the present study was designed to evaluate the effects of two different concentrations of TCA as a hemostatic agent on bond strength of a kind of self-etch adhesive to enamel and dentin.

The null hypothesis of this study was that the resin-enamel/dentin bond strength of self-etch adhesive systems is not different from those achieved when the material is applied to enamel and dentin treated with TCA.

## MATERIALS AND METHODS

Thirty-six human third molars were used in the present *in vitro* study. The teeth were cleaned of any residual tissue tags, pumiced, and washed under tap water immediately after extraction. The teeth were stored in distilled water at +4°C for less than a week. The roots were removed at CEJ with a slow-speed diamond saw under copious water spray. The crown of each sample yielded two tooth segments measuring approximately 7 × 8 mm; therefore, 72 enamel and dentin samples were produced. Subsequently, the samples were mounted with the buccal and lingual surfaces upward in a plastic holder using self-curing acrylic resin. The buccal surfaces of the samples were flush with the acrylic resin to form one flat surface.

In the enamel groups the central part of the embedded sample was ground flat with 320- and 400 grit silicon carbide papers, respectively. In the dentinal groups the surfaces were abraded to expose superficial dentin; the exposed dentin surface was then ground flat with the use of 320- and 400 grit silicon carbide papers, respectively.

The enamel and dentin samples were randomly assigned to three groups:

- Group I (E1): Control; application of Clearfil SE Bond (CSEB) according to the instructions provided by the manufacturer.
- Group II (E2): Application of 35% TCA before applying Clearfil SE Bond.
- Group III (E3): Application of 50% TCA before applying Clearfil SE Bond.
- Group IV (D1): Control (application of Clearfil SE Bond).
- Group V (D2): Application of 35% TCA before applying Clearfil SE Bond.
- Group VI (D3): Application of 50% TCA before applying Clearfil SE Bond.

### Bonding Procedure

A piece of adhesive tape with a hole, measuring 4 mm in diameter, was firmly adapted to the center of the flattened part of the enamel and dentin surfaces to mark the bonding surface. The self-etch adhesive system Clearfil SE Bond

**Table 1:** Shear bond strength ± SD of a self-etching adhesive to the normal and TCA-treated enamel and dentin (MPa)

Enamel groups	Mean ± SD	Dentin groups	Mean ± SD
E1	34.73 ± 5.66 <sup>a</sup>	D1	20.94 ± 9.84 <sup>b</sup>
E2	35.32 ± 7.38 <sup>a</sup>	D2	19.32 ± 6.20 <sup>b</sup>
E3	23.75 ± 9.67 <sup>cb</sup>	D3	23.50 ± 6.63 <sup>b</sup>
p-value = 0.001		p-value = 0.425	

Groups with the same superscript are not statistically different ( $p > 0.05$ ); E: Enamel; D: Dentin

(Kuraray; Osaka, Japan) was applied to the delineated surface based on manufacturer's instructions. One drop of the acidic primer was applied to the enamel and dentin surfaces for 20 seconds and gently air dried. A layer of bonding resin was applied with a microbrush, spread gently with air, and light cured for 10 seconds using a light-curing unit with an intensity output of 480 mW/cm<sup>2</sup> (Coltolux 2.5, Coltene AG, Feldwiesenstrasse Altstätten/Switzerland). A plastic mold with a circular hole (4 mm in external diameter, 2 mm in internal diameter and a height of 1 mm) (Orthorings, Ortho organizers Inc, CA, USA) was placed and fixed over the hole in the adhesive tape. A composite resin (Z100, 3M ESPE, St. Paul, MN, USA) layer was placed and cured in the mold, forming cylindrical posts at right angles to the surfaces. Each specimen was thoroughly cured for 40 seconds. After retrieval from the plastic molds, the specimens were stored in distilled water at 37°C for 24 hours. Subsequently, the samples underwent a 500-round of thermocycling procedure at 5°C/ 55°C, with a dwell time of 30 seconds (MP Based, KARA 1000, Tehran, Iran).

The shear bond strength (SBS) values were measured with a DARTEC Universal Testing Machine (Model HC10, Stourbridge, England). A knife-edge shearing rod was used at a crosshead speed of 1 mm/min. The distance from the probe to the enamel or dentin surface was monitored using a spacer consisting of two celluloid matrix bands. The SBS values of the specimens were calculated and expressed in MPa. Then, fracture analysis of the bonded surface was performed under a stereomicroscope (MBC 10, St. Petersburg, Russia) at ×32 magnification. Failures were classified as adhesive (more than 75% of failure between tooth and restorative material), cohesive (more than 75% of the failure within the restorative material), or a mixture of the two types mentioned above. Statistical analysis was carried out using SPSS 11.5 software. The SBS data of the groups were statistically analyzed with one-way ANOVA and Scheffé's test. Chi-squared test was used to compare the fracture modes of the samples. Statistical significance was defined at  $p = 0.05$  for all the tests.

## RESULTS

SBSs in MPa (mean ± SD) of the groups under study are presented in Table 1. One-way ANOVA revealed significant differences only in bond strength values of the enamel groups ( $p = 0.001$ ), with no significant differences between dentin groups ( $p = 0.425$ ).

According to the Scheffé's test, in the enamel groups the samples treated with 50% TCA gel (E3) exhibited significantly lower bond strength values compared to those of the control group samples ( $p = 0.006$ ); no significant differences were observed between E1 and E2 groups ( $p = 0.983$ ).

The fracture modes of groups are summarized in Table 2. In each group, approximately 30% of the samples exhibited the mixed fracture mode. E3 group samples had more numerous adhesive failures, with the lowest mean of bond strength values. Although significant differences were observed between the three enamel groups in relation to fracture modes ( $p < 0.05$ ), no significant differences were found between the three dentin groups in this respect ( $p > 0.05$ ).

## DISCUSSION

In this study, the effects of 35% and 50% TCA on bond strength of one self-etch adhesive to enamel and dentin were evaluated. The results showed that contamination/conditioning of enamel surface with 35% TCA before application of CSEB does not adversely affect bond strength; however, 50% TCA gives rise to a decreased bond strength of the adhesive to enamel despite the fact that it is sufficient for bonding of composite resins to enamel. In addition, the results showed that previous contact of 35% or 50% TCA with dentin before application of Clearfil SE Bond does not significantly influence SBS in comparison to the control group.

The results of the present study in relation to two-step self-etch adhesives confirmed the results of two previous studies on etch-and-rinse adhesives.<sup>21,22</sup> In the present study,

**Table 2:** Different fracture modes in the study groups N (%)

Groups/mode of fracture	Adhesive	Cohesive (enamel/ dentin)	Mixed	Total
1. Clearfil SE Bond on enamel (control)	4 (33.3%)	3 (25%)	5 (41.7%)	12 (100%)
2. TCA 35% plus Clearfil SE Bond-enamel	3 (25%)	4 (33.3%)	5 (41.7%)	12 (100%)
3. TCA 50% plus Clearfil SE Bond-enamel	7 (58.3%)	1 (8.4%)	4 (33.3%)	12 (100%)
4. Clearfil SE Bond-dentin (control)	7 (58.3%)	1 (8.4%)	4 (33.3%)	12 (100%)
5. TCA 35% plus Clearfil SE Bond-dentin	6 (50%)	2 (16.7%)	4 (33.3%)	12 (100%)
6. TCA 50% plus Clearfil SE Bond-dentin	4 (33.3%)	1 (8.4%)	7 (58.3%)	12 (100%)

TCA: Trichloroacetic acid; CSEB: Clearfil SE Bond

the mean of enamel bond strength of CSEB two-step self-etch adhesive in the control group was approximately 34 MPa. In addition, dentin bond strength value with this adhesive was approximately 20 MPa. Recently, CSEB adhesive has been suggested as a gold standard for dentin adhesives with proper and sufficient bond strength for cut enamel without acid preconditioning.<sup>23</sup>

Erickson et al reported that although chemical bonding might be provided by some self-etch adhesive systems, it does not sufficiently increase bond strength to compete with the bond produced by etch-and-rinse systems, which use phosphoric acid to etch the enamel.<sup>24</sup> Based on data available, one of the disadvantages of self-etch adhesives is their deficient bonding capacity to enamel; therefore, it is recommended to etch enamel before application of the systems mentioned above in order to create more consistent resin tag-like penetrations.<sup>25,26</sup> Calcium and phosphate released during enamel decalcification lead to a gradual cessation of acidic primer activity, resulting in a lower bond strength of self-etch adhesives to enamel.<sup>19</sup>

In the present study, 50% TCA had a detrimental effect on the bond of self-etch adhesive to enamel. pH value of TCA is below 1, which is comparable to that of phosphoric acid. Self-etch adhesives are divided into three groups of mild, moderate and strong based on their acidity.<sup>23</sup> CSEB is a two-step self-etch adhesive with a pH value of 2.5; it is considered a mild self-etch adhesive. Recently, a study showed the efficacy of the etching effect of 50% TCA on cut enamel through SEM and bond strength evaluations.<sup>21</sup>

In the present study, application of 35% TCA in group E2 did not result in statistically significant effects on bond strength of the adhesive evaluated. It seems removing large amounts of calcium and phosphate ions from the enamel surface to produce a highly demineralized surface by the acid results in better penetration of acidic primer. Conversely, it seems that application of 50% TCA in group E3, interfered with penetration of self-etch primer into enamel, resulted in decreased bond strength. In addition to the adverse effect of this concentration of TCA on enamel surface,<sup>21</sup> one reason for this decrease might be the by-products produced during application of 50% TCA to the enamel surface. These by-products might not be removable, might remain on enamel surfaces and might interfere with acidic primer and penetration of functional monomers.

In the present study, since the acidic primer of CSEB was also applied to enamel after etching with phosphoric acid, it might have resulted in the dissolution and detachment of more hydroxyapatite crystals from the surface, resulting in a state called overetching of surface; in such cases, since rinsing is not carried out after application of the acidic primer the bond of the second part of the adhesive is sometimes

formed with loose hydroxyapatite crystals, resulting in a decreased bond strength. However, in the case of 35% TCA, it is possible that the lower concentration of the acid has resulted in the removal of fewer crystals; as a result, a better bond has formed. However, further studies are required on the subject.

On the other hand, the results of present study are consistent with the results of a previous study in relation to the fact that application of 35% TCA on enamel substrate as a conditioning agent did not have a detrimental effect on bond to enamel but 50% TCA decreased enamel bond strength of the self-etch adhesive under study.<sup>21</sup> In other words, it is necessary that studies in future evaluate the effect of TCA preconditioning with phosphoric acid on bond strength to cut and uncut enamel with the use of self-etch adhesives.

Some previous studies have demonstrated the positive effect of preconditioning of enamel with phosphoric acid on the bond strength of self-etch adhesives.<sup>27,28</sup> If TCA plays the role of an appropriate preconditioning agent in addition to its hemostatic effect, it appears the dental practitioner will not need a separate acid-etching step with phosphoric acid in cases in which there is a need for its hemostatic effect.

Some previous studies have confirmed the reliable results of etching with phosphoric acid to produce a bond with enamel before the application of a self-etch adhesive.<sup>23,27,28</sup> In the present study, application of TCA was evaluated from two aspects: the first one was the possibility of its contact with the substrate for bonding of the self-etch adhesive, which is in fact its use as a preconditioning agent. The second aspect was its use as a hemostatic agent to control hemorrhage, gingival secretions and saliva, during which it inadvertently contacts tooth structures. It is necessary to discuss these matters separately for enamel and dentin substrates.

Regarding the hemostatic effect, application of TCA did not have any detrimental effect on the dentin bond strength of self-etch adhesive; as such, the bond strength values did not exhibit any differences with the use of both 45 and 50% concentrations of TCA in comparison with the control group. The authors of the present study evaluated and compared the hemostatic effects of 35, 50 and 90% concentrations on some volunteers who needed cervical composite resin restorations in a preliminary study. The results did not reveal any differences in the hemostatic properties between the three concentrations. However, in relation to the conditioning effect, the two concentrations of TCA did not have any detrimental effects on dentin bond strength and even the 50% concentration increased bond strength, although the difference was not statistically significant. If

future studies reveal better clinical results with 50% TCA in relation to the control of hemorrhage and gingival secretions, it appears the use of 50% TCA in cavities with predominantly dentin substrate or with weak enamel margins can be better recommended.

Recently, a study showed improvements in bond strength values of an etch-and-rinse adhesive to enamel with the application of both 35 and 50% concentrations of TCA. On the other hand, some previous studies have shown decreased bond strength of composite resins to tooth structures with the use of other hemostatic agents.<sup>6,7,29</sup> It is of clinical interest that some materials with acidic pH values produce deposits on tooth structures, which might interfere with the bonding process.<sup>21</sup> For example, Kuphasuk et al showed remnants of aluminum chloride in tooth structures and decreased bond strength in their study.<sup>5</sup>

In the present study, there were no significant differences between SBS values of dentin groups. Moreover, frequency of adhesive failures decreased with 50% TCA, while mixed failures increased. The results of SBS test were consistent with the results of some previous studies.<sup>4,30,31</sup> Some studies have confirmed that dentin contamination with different hemostatic agents does not reduce bond strength. Two studies reported no decrease in dentin bond strength of resin cements to dentin with the use of an aluminum chloride-containing astringent.<sup>30,31</sup> In addition, they concluded that because of the demineralizing effect of  $AlCl_3$ , the etching effect of the self-etch primer is enhanced.<sup>4</sup> Kimmes et al showed that application of Viscostat or Viscostat Plus as hemostatic agents has no adverse effect on the SBS values of composite resin to dentin using Optibond Solo Plus.<sup>30</sup> If this material is not removed prior to the bonding process, bond strength will compromise; while removing Viscostat using copious amounts of water before the bonding process greatly increases bond strength.<sup>1</sup> It is believed that the acidity of the hemostatic agent has a role in changes in the dentin surface. By increasing the contact duration, changes in the dentin surface will be more widespread, from removal of the smear layer to opened dentinal tubules and etching of the underlying dentin.<sup>7,32</sup>

In the present study, the frequency of adhesive fracture in group E3 (50% TCA) increased, which is consistent with the relevant bond strength outcomes. It seems logical that when acidic primer has decreased penetration into the enamel structure, forces will be concentrated in the weakest part which is the tooth-adhesive interface, resulting in lower bond strength in group E3.

Finally, it should be pointed out that in the present study the effect of two different concentrations of TCA as a hemostatic/preconditioning agent on the enamel bond of only one type of two-step self-etch adhesive was evaluated.

It is obvious that it is necessary to evaluate different kinds of adhesives with different chemical compositions and functional agents. In addition, the effects of different concentrations of TCA as a hemostatic agent have not been evaluated in clinical situations, necessitating accurate evaluations in future.

## CONCLUSION

Under the limitations of the present study it was concluded that use of 35% and 50% concentrations of TCA did not adversely affect the dentin bond of two-step self-etch adhesive. Moreover, application of 35% TCA did not have any adverse effect on enamel bond strength of the same adhesive.

## CLINICAL SIGNIFICANCE

In cervical cavities with enamel or dentin margins, restored with two-step self-etch adhesives and composite resin, 35% TCA can be used as a hemostatic agent without any adverse effects on the bond strength. Application of 50% TCA in cavities with an enamel substrate is not recommended.

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## ABOUT THE AUTHORS

### Kamyar Fathpour

Assistant Professor, Department of Operative Dentistry, Torabinejad Dental Research Center, Isfahan University of Medical Sciences Isfahan, Iran

### Maryam Khoroushi (Corresponding Author)

Associate Professor, Dental Materials Research Center and Department of Operative Dentistry, Isfahan University of Medical Sciences Isfahan Iran, Phone: +98 311 6687080, e-mail: khoroushi@dnt.mui.ac.ir