

## Original Article

## Effect of Etching Time and Preparation on Push-Out Bond Strength of Composite to Intracanal Dentin of Primary Anterior Teeth

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### Abstract

**Objectives:** This in-vitro study aimed to compare the push-out bond strength of composite to prepared and unprepared intracanal dentin using a 5th generation bonding agent in primary anterior teeth after etching for seven and 15 seconds.

**Materials and Methods:** Sixty primary anterior teeth were randomly divided into four groups. In groups one and two, intracanal dentin remained intact while in groups three and four it was prepared using a #5 round bur and low-speed handpiece. Single Bond 2 was used in groups one and three after 15 seconds and in groups two and four after seven seconds of etching with phosphoric acid. After restoring with composite resin and incubation, the specimens were subjected to push-out bond strength test. Data were analyzed using two-way ANOVA and Kaplan Meier curves.

**Results:** The mean bond strength was not significantly different between seven and 15 seconds etching times ( $P=0.198$ ). Dentin preparation had no significant effect on the mean bond strength ( $P=0.838$ ). The interaction effect of etching time and dentin preparation was not significant either ( $P=0.680$ ).

**Conclusions:** Decreasing the etching time from 15 to seven seconds and preparation of intracanal dentin had no significant effect on push-out bond strength of composite to intracanal dentin of primary anterior teeth.

**Keywords:** Dental Etching; Composite Resins; Dentin; Tooth, Deciduous

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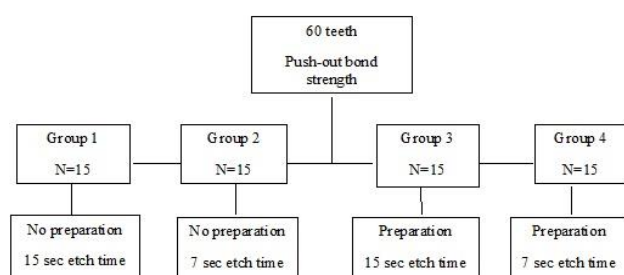
### INTRODUCTION

Maintaining primary anterior teeth in a developing dental arch is imperative [1-5]. Early loss of primary teeth in young children can impair mastication, decrease vertical facial dimension, cause parafunctional habits, lead to space loss, compromise esthetics and interfere with social and personality development of children. Although preventable, dental caries is among the most common chronic diseases in children [1].

Early childhood caries is a common condition of childhood and results in rapid destruction of primary maxillary anterior teeth [2]. Before the introduction of dentin bonding agents, extraction was the only available solution for the severely

damaged primary anterior teeth due to their difficult restoration [1-5]. The American Academy of Pediatric Dentistry in its guideline on restorative dentistry recommends strip crowns, open face stainless steel crowns and pre-veneered stainless steel crowns for full coverage restoration of primary teeth; however, application of these modalities requires adequate amount of coronal tooth structure [6]. Therefore, restoration of teeth with severe caries affecting the entire crown at an early age is challenging; this topic has not been paid much attention in the literature.

Caries removal in severely damaged teeth often results in pulp exposure, necessitating pulpectomy. With recent advances in dentin



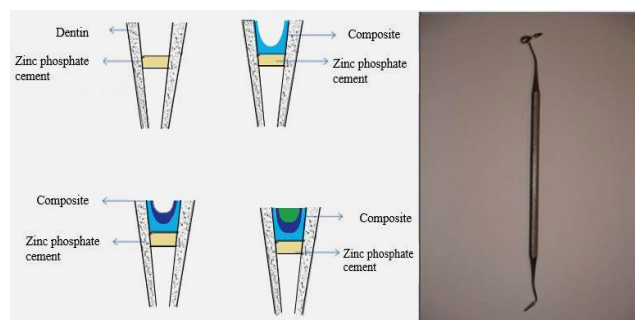
**Fig.1:** Distribution of 60 extracted primary anterior teeth into four groups for the study of push-out bond strength

adhesives, clinicians can build up the whole crown with composite resin by obtaining retention from the coronal 3 mm of the root canal using dentin bonding agents [1-3,5,7].

Intracanal posts can be fabricated of different materials such as composite resin, metal, biological materials [1,5], reinforced fiber, fiberglass [5], orthodontic wires and omega-shaped stainless steel wires [7]; of the aforementioned materials, composite posts are more commonly used for primary teeth [8].

Most studies on the bond strength of dentin adhesives have reported lower bond strength to primary compared to permanent teeth [9]. Different properties of primary and permanent dentin may explain the difference in bond strength values [9,10]. On the other hand, it has been shown that application of the same etching time of permanent dentin for primary dentin decreases resin bond strength to primary dentin [10]. Scanning electron microscopic analyses revealed that application of similar etching time for primary and permanent dentin resulted in deeper demineralization of primary dentin by 25-30% compared to permanent dentin [9,11]. Resin monomers cannot penetrate into the depth of demineralized area. Consequently, an area composed of denuded collagen fibrils remains, which would be the weakest zone at the adhesive-dentin interface [9].

It should be noted that previous studies in this



**Fig.2:** Composite was packed using a composite condenser instrument

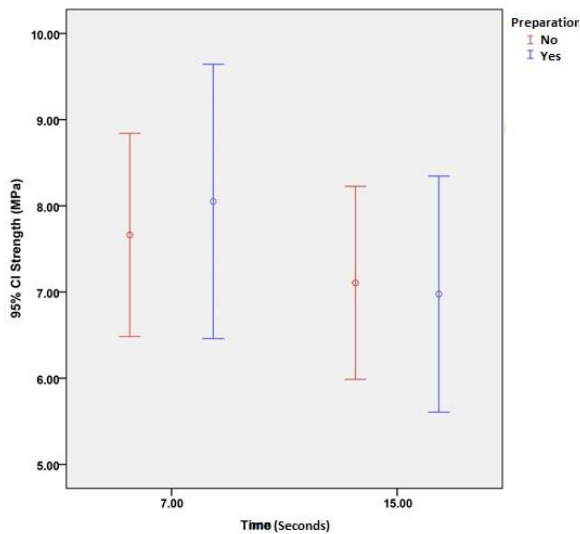
regard mainly assessed dentin far from the dental pulp (adjacent to dentinoenamel junction). Moreover, number of studies on the effect of etching time on bond strength of primary intracanal dentin is limited. Results of a previous study revealed that bond strength to primary intracanal dentin was not significantly different for the 5th, 6th and 7th generation bonding agents following a 15-second etching time, and the obtained bond strength values were all lower than those in permanent teeth [12].

Concerning the short length of intracanal posts (3mm) recommended for the buildup of primary tooth crowns [2], it is important to obtain maximum retention from the coronal 3mm of the root canal to further ensure the clinical success of these restorations.

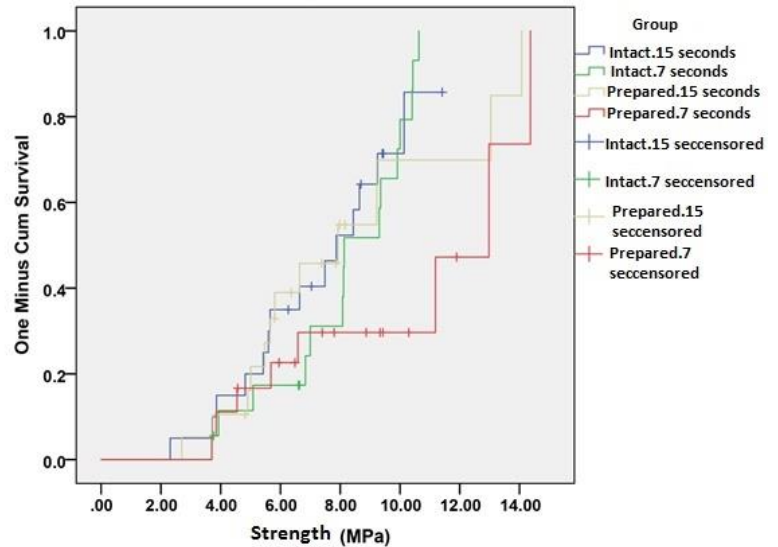
Preparation of intracanal dentin when preparing a post space is feasible after pulpectomy and root canal filling. Considering the limited number of studies on optimal etching time of primary intracanal dentin, this study sought to assess the effect of seven versus 15 seconds of etching and preparation versus no preparation of intracanal dentin on its push-out bond strength to composite using a 5th generation adhesive system (Single Bond 2) in primary anterior teeth.

## MATERIALS AND METHODS

The study protocol was approved in the committee of medical ethics of Tehran University of Medical Sciences (code:5080).



**Fig.3:** Error bar of the mean and 95% confidence interval of push-out bond strength in the two groups of unprepared and prepared dentin after seven and 15 seconds of etching



**Fig.4:** One minus Kaplan-Meier survival functions for the bond strength of the study groups considering cohesive failures as censored data

Written informed consent was obtained from the parents or legal guardians of children whose extracted teeth were used in this study. A total of 60 primary anterior teeth extracted within the past six months due to severe caries were evaluated in this experimental study. Teeth that met the following inclusion criteria were selected: Root resorption by no more than half the root length, a minimum of 6 mm of root length remaining and absence of internal root resorption in the coronal third of the root canals. The teeth were immersed in 0.5% chloramine T solution (Merck, Darmstadt, Germany) for one week, transferred to distilled water and stored at 4°C. Tooth crowns were cut one millimeter above the cemento-enamel junction with a diamond disc perpendicular to the long axis of the teeth. The teeth were then randomly divided into four groups of 15 and coded in a blind fashion (Fig. 1).

Group 1 specimens received no tooth preparation and were subjected to 15 seconds of etching with phosphoric acid. After rinsing and drying with cotton pellet, Single Bond 2 (3M ESPE, St. Paul, MN, USA) 5th generation bonding agent was applied according to the manufacturer's

instructions.

Group 2 specimens received no tooth preparation and were subjected to seven seconds of etching with phosphoric acid. After rinsing and drying with cotton pellet, Single Bond 2 5th generation bonding agent was applied according to the manufacturer's instructions.

Group 3 specimens received dentin preparation and were subjected to 15 seconds of etching with phosphoric acid. After rinsing and drying with cotton pellet, Single Bond 2 5th generation bonding agent was applied according to the manufacturer's instructions.

Group 4 specimens received dentin preparation and were subjected to seven seconds of etching with phosphoric acid. After rinsing and drying with cotton pellet, Single Bond 2 5th generation bonding agent was applied according to the manufacturer's instructions.

Root canals were instrumented by K-files (Mani Inc., Tokyo, Japan) to three sizes larger. In groups 3 and 4, dentinal canal walls were prepared by 0.5mm using a #5 carbide round bur (Kerr, Orange, CA, USA) and a low-speed handpiece (NSK, Tokyo, Japan). Canals were rinsed with saline solution and dried with paper

**Table 1:** The mean and standard deviation of push-out bond strength (MPa) in unprepared and prepared primary anterior teeth after seven and 15 seconds of etching

Preparation	Time (s)	Mean strength±standard deviation
No	15	7.10±2.39
No	7	7.66±2.37
Yes	15	6.97±2.84
Yes	7	8.05±3.19

points (VDW GmbH, Munich, Germany). Canals were not filled with the materials commonly used for the filling of pulpectomized teeth since it had no effect on the results. Next, one layer of zinc phosphate cement (Harvard, Hoppegarten, Germany) with 1mm thickness was applied in such a way to obtain a proper apical seat for condensation of composite. Coronal one-third of the canals was then filled with a posterior composite resin (Filtek P60, 3M ESPE, St. Paul, MN, USA). Composite was incrementally applied by a composite condenser instrument and the root canal was filled with three increments of composite (Fig. 2). Each layer was cured for 20 seconds. All samples were light cured (Wood Pecker, Guangxi, China) under similar conditions in terms of the LED light-curing unit used (Foshan Cicada light tester, Guangdong, China), the light intensity (800 mW/cm<sup>2</sup>) and the distance of 2mm from the tooth surface. Next, all samples were immersed in distilled water and stored in an incubator at 37°C (MalekKhosravi, Tehran, Iran) for 24 hours. Samples were then mounted in clear acrylic resin (Azar Material Processing Company, Tehran, Iran) and a one-millimeter thick section was made at the middle of the prepared area using a Mecatome (Model T201A; Persi, Paris, France). The push-out bond strength test was performed using a universal testing machine (Z05, Zwick/ Roell, Ulm, Germany). The load was applied to the bonding interface at a crosshead speed of 0.5 mm/minute in an apico-cervical direction using a cylindrical stainless steel plunger measuring 1.4mm in

diameter and 5.2mm in height. The load at fracture (debonding) was recorded in Newtons (N). The load at fracture (N) was divided by the cross-sectional area (mm<sup>2</sup>) to calculate stress at the fracture point and report the bond strength in Megapascals (MPa). Before performing the push-out test, photographs were captured by a digital camera (Canon, Eos 600D, Tokyo, Japan) from the two sides of the sectioned specimen. Photographs were imported to Auto CAD version 2013 software (Cadapult Software Solutions, Inc., OR, USA) and the composite-dentin interface (border) was marked on the photographs. The areas at the two sides of the interface were then outlined by the software and the cross sectional area was calculated using the formula below:

$$(A1+A2)\frac{h}{2}$$

Where A1 is the circumference of one side, A2 is the circumference of the other side and h is the height of the prepared section of the root in millimeters. After the conduction of push-out test, samples were examined under a light microscope at x40 magnification (SZX2-zb16; Olympus, Tokyo, Japan) to determine the mode of failure. The mode of failure was categorized into four groups of adhesive, cohesive in dentin, cohesive in composite and mixed. The data were recorded and the effect of etching time and type of dentin (prepared or unprepared) on the push-out bond strength was evaluated using two-way ANOVA, log-rank test and the Kaplan Meier curves. P≤0.05 was considered statistically significant.

## RESULTS

Data analysis with two-way ANOVA revealed that the mean bond strength was not significantly different following seven and 15 seconds of etching (P=0.198). Dentin preparation had no significant effect on bond strength either (P=0.838). The interaction effect of etching time and preparation on bond strength was not significant (P=0.680) (Table 1 and Fig. 3).

**Table 2:** Failure modes of composite bond to unprepared and prepared intracanal dentin of primary anterior teeth after seven and 15 seconds of etching

Groups	Cohesive in dentin N(%)	Cohesive in composite N(%)	Mixed N(%)	Adhesive N(%)
Intact/15 seconds	1(5)	5(25)	7(35)	7(35)
Intact/7 seconds	1(5.6)	2(11.1)	12(66.7)	3(16.7)
Prepared/15 seconds	2(10.5)	5(26.3)	9(47.4)	3(15.8)
Prepared/7 seconds	2(11.1)	8(44.4)	6(33.3)	2(11.1)

Data analysis with log-rank test on Kaplan Meier curves by taking into account the mode of failure showed no significant difference among the four groups (P=0.303, Fig. 4 and Tables 2 and 3).

**DISCUSSION**

Several factors affect the bond strength to dentin in vitro such as type and age of tooth, degree of dentin mineralization, bonded surface area, type of bond strength test (shear or tensile), storage medium of the tooth, relative humidity of the substrate and testing conditions. These factors may explain the controversies in the bond strength test results of different studies [13,14]. Push-out test was performed in the current study, which applies shear load to the composite-bonding agent and dentin-bonding agent interfaces. The push-out test results are closer to the subclinical results compared to the linear shear test results [15]. It should be noted that the root canals in our study were not filled with calcium hydroxide, zinc oxide eugenol or iodoform-containing pastes in order to eliminate their possible confounding effects on the results. By doing so, the effects of etching time and presence/absence of dentin preparation on bond strength were exclusively evaluated.

On the other hand, we applied composite resin in three increments to the root canal using a condenser designed for this purpose. By doing so, we minimized the ratio of bonded to unbonded surfaces especially in the first layer (over the adhesive-dentin interface) to subsequently minimize the configuration factor and consequently the polymerization shrinkage stress of composite and increase the bond

strength. As stated earlier, type of tooth (primary/permanent) affects the bond strength. Studies on push-out bond strength have reported higher bond strength values in permanent than in primary teeth [16]. Greater amount of available inter-tubular dentin substrate in permanent compared to primary teeth for bond to adhesive explains the difference in bond strength of primary and permanent dentin [9]. Despite limited number of studies on factors affecting the push-out bond strength of primary teeth [12], several studies have evaluated the bond strength to primary dentin far from the dental pulp using Single Bond and reported tensile bond strength higher than the values obtained in the current study [9,17]. The greater density and diameter of the dentinal tubules near the dental pulp [18] explains the higher bond strength in the aforementioned studies since they evaluated dentin far from the dental pulp (due to greater amount of substrate for bonding following etching) [9].

**Table 3:** The mean and median of push-out bond strength (MPa) in unprepared and prepared primary anterior teeth after 7 and 15 seconds of etching using Kaplan-Meier curve

Group	Mean <sup>a</sup>		Median	
	Estimate	Standard error	Estimate	Standard error
Intact/15 seconds	7.606	.620	7.870	1.168
Intact/7 seconds	8.189	.554	8.131	1.119
Prepared/15 seconds	8.509	1.056	7.925	1.443
Prepared/7 seconds	10.653	1.089	12.978	2.996
Total	8.565	.429	8.438	.611

a. Estimation is limited to the largest survival time if it is censored

Considering etching time and the differences in some micromechanical and histological properties of primary and permanent dentin [17], our alternative hypothesis was that applying shorter etching time for primary dentin than that recommended by the manufacturers for permanent dentin would yield higher bond strength values. This hypothesis has been approved in some previous studies [9-11,13,17,19,20]. Evidence shows that decreasing the etching time of primary dentin increases the quality [14] and uniformity [10] of mechanical interlocking of resin tags between collagen fibrils in the hybrid layer. By decreasing the etching time, the morphology of the etched surface of primary dentin would be similar to that of permanent teeth after etching [13]. Application of shorter etching time prevents the formation of a layer with partially penetrated resin tags and denuded collagen fibrils beneath the hybrid layer [9,11], since this layer would be the weakest area at the dentin-adhesive interface [9]. However, the results of the current study did not reveal a significant difference in this regard and our alternative hypothesis was refuted. A previous study reported results similar to ours and did not find any significant difference in bond strength following seven and 15 seconds of etching [9]. But, it should be noted that in contrast to the current study, the afore-mentioned studies had been conducted on permanent dentin and used bond strength tests other than the push-out test. Such variability in methodologies as well as other factors such as the bonded surface area of dentin might have affected the results and may be responsible for the differences in the results of studies [13,14]. With regard to preparation of dentin surface, it should be noted that dentin has a tubular structure, and density and diameter of dentinal tubules closer to the pulp are greater than those in areas closer to the dentinoenamel junction. Dentinal tubules closer to the pulp have less calcified tissue. Thus, the bonding substrate is more abundant farther from the pulp and

towards the dentinoenamel junction [10]. Therefore, our second alternative hypothesis was that bond strength would increase by preparing the intracanal dentin (getting closer to the cementodentinal junction). However, our results showed that bond strength was not significantly different in groups with and without dentin preparation. This finding may be attributed, at least partly, to the difference in the structure of coronal and radicular dentin because radicular dentinal tubules are less divergent than coronal dentinal tubules [21] and thus, less structural changes occur in radicular dentin as moving from the pulp towards the cementodentinal junction. Therefore, insignificant effect of radicular dentin preparation on bond strength was somehow expected. With regard to failure mode, a previous study showed that failure mode in primary dentin was often of adhesive or mixed type [9]. However, assessment of failure mode in our four groups showed that the frequency of cohesive mode of failure (within composite or dentin) was significantly higher (55%) in group four with seven seconds of etching and dentin preparation compared to other groups.

Also, group four had the lowest frequency of adhesive failure compared to the other groups. Analysis of these results in our study showed that the mean and median of bond strength in group four were significantly higher than those in the remaining three groups, indicating that despite the insignificant independent effects of decreasing the etching time and dentin preparation on bond strength, combination of these two factors may increase the bond strength. Considering the role of poly alkenoic acid in the formulation of Single Bond [17], which results in carboxylate cross linking between the carboxyl groups in the bonding agent and calcium in dentin hydroxyapatite [22], a hypothesis was suggested that the quality of hybrid layer can be enhanced by decreasing the etching time of primary dentin and maximizing micromechanical interlocking of resin tags

between collagen fibrils [14]. Moreover, further preparation of dentin (taking into account the thickness of radicular walls) provides more mineral substrate for chemical bond to poly alkenoic acid present in Single Bond. By taking these measures, higher bond strength can be expected in future studies using 5th generation dentin bonding agents containing poly alkenoic acid.

## CONCLUSION

Within the limitations of this in-vitro study, the results showed that seven and 15 seconds of etching with phosphoric acid and dentin preparation had no significant effect on bond strength of intracanal primary dentin to composite posts. When combined with dentin preparation, etching for seven seconds may yield more favorable bond strength.

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