



Effect of Different Etching Time on Bond Strength of Composite Resin to Dentin

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Authors' contributions

This work was carried out in collaboration between all authors. Author NM designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors IB and RS managed the analyses of the study. Author PM managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Aim: The aim of this study is to evaluate the bond strength of composite resin to dentin surface with various etching protocol.

Methodology: Three sample groups, each consisting of ten permanent mandibular first molar teeth were established. The buccal surfaces of the samples were grinded with the help of straight fissured diamond abrasive removing enamel layer. Samples of Group A, B and C were etched for 15, 30 and 60 seconds respectively, with 37% phosphoric acid. All the samples were washed for 1 min and then dried. Samples were bonded for 15 seconds and restored with composite resin and cured. Samples were subjected under universal testing machine for bond failure.

Results: Bond strength was found to be much higher in group B than in group C and A with a confidence of more than 98.828% (P value: 0.0675).

Conclusion: Within the limitations of this *in vitro* study; it can be concluded that 30 seconds of etching time gave better bond strength due to longitudinal tubules which influenced the dentin hybridization process in the sense of forming more resin tags that, in turn, contributed to bond strength.

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1. INTRODUCTION

Over the years, phosphoric acid etching has become the standard procedure for bonding to improve the surface characteristics before the application of adhesive bonding agents and fissure sealant [1]. The penetration of adhesive resin monomers into the porous zone results in the formation of resin tags, thereby establishing micromechanical interlocking within the etched surface. Therefore, regardless of the adhesive system, use of phosphoric acid supports achievement of a strong and durable bond [2]. Phosphoric acid etching gel is applied to dentin substrate to remove the smear layer. After rinsing, the dentin surface becomes demineralized with exposure of the collagen fibers. This would leave collagen fibers exposed and susceptible to hydrolysis, which may weaken the bonding [3]. Thus, it is speculated that as shallow a demineralization as possible might give the adhesive system a better chance to diffuse into the entire collagen network [4]. For that, acid-etching time of 15 seconds has been suggested by various authors, aiming at an adequate bond to normal dentin. Dentin hybridization is a modern dental adhesion procedure, which was first described by Nakabayashi et al. [5]. Hybridized dentin begins under the dentin surface after surface and subsurface demineralization and adhesive monomer infiltration into exposed collagen network [6]. Thus, the result of this revolutionary method, discovery by Nakabayashi and colleagues has opened new horizons of restorative dentistry. Over the years, phosphoric acid etching has become the standard procedure for adhesive dentistry. In 1954 Buonocore introduced acid etching procedure as a pre-treatment method that enhances the bonding strength of composite resins for the first time [7]. Its clinical application has been presented in 1976 by Cueto and Buonocore []. The dentin surface becomes demineralized with exposure of the collagen fibers [8]. To obtain adequate resin-dentin bonding, resin monomers must penetrate this demineralized surface dentin in order to produce hybridization. The clinical success of restorative material depends upon a good adhesion with dentinal surface so as to resist various dislodging forces acting within the oral cavity [9].

The need for restorative material, with better bond characteristics and strength to withstand

the stress of masticatory forces, leads to the recent advances in the restorative dentistry. The composite resins are one of the commonest restorative materials used now-a-days due to its aesthetic appearance but they have some imperfections such as polymerization shrinkage [10].

The present study was done to evaluate the bond strength of universal sub micron hybrid composite resin BRILLIANT EverGlow-Coltene, to dentin surface with various etching protocol.

2. MATERIALS AND METHODS

A total of 30 extracted human permanent mandibular molar teeth with neither carious lesions nor restorations, were selected for this *in vitro* study. Each tooth underwent scaling and root planning with an ultrasonic device to remove residual organic tissues. Then, the teeth were immersed in 2.5% sodium hypochlorite solution and rinsed with running water for 10 min.

Acrylic blocks were prepared by cold cure acrylic resin material. The selected molars were embedded into the blocks. The blocks were then put in water to avoid expansion of the material. The buccal surfaces of the samples were grinded with the help of straight fissured diamond abrasive up to 3 mm depth of the dentinal surface.

Dentinal surfaces were acid etched with 37% phosphoric acid into following groups:

- Group A: 15 seconds
- Group B: 30 seconds
- Group C: 60 seconds

Etching was done to make the surface smear free. Samples were then bonded with One coat Bond SL by Coltene and light cured with LED light for 20 seconds and restored with BRILLIANT-EverGlow-Coltene submicron hybrid composite light cure composite resin. The specimens were stored in distilled water for 24 hrs.

Specimens were then transferred to the Universal testing machine with a crosshead speed of 0.5 mm/min until fracture with tip diameter 1.5 mm. It was subjected to compressive test determination which created buckling of the restoration. It resulted in formation of a tensile stress in the dentinal walls.

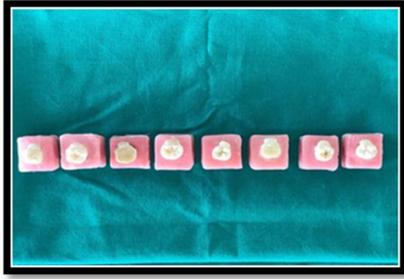


Fig. 1. Acrylic mould prepared for each samples

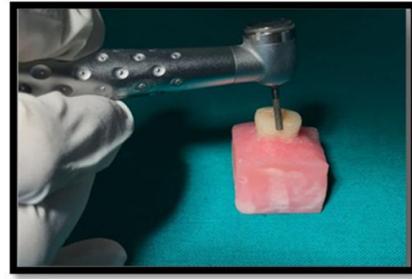


Fig. 2. The buccal surfaces of the samples grinded with the help of straight fissured diamond abrasive up to dentin surface



Fig. 3. 37% Phosphoric acid

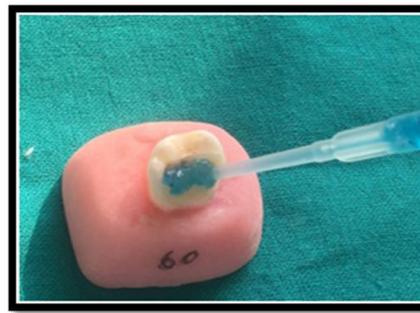


Fig. 4. Etching of dentin surface



Fig. 5. Application of bond

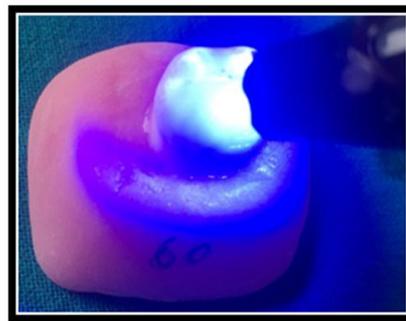


Fig. 6. Curing after application of bond

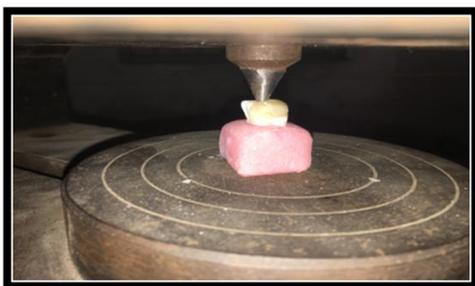


Fig. 7. Specimens transferred to the UTM with a crosshead speed of 0.5 mm/min until fracture with tip diameter 1.5 mm



Fig. 8. Universal testing machine

The load required to debond the specimen was recorded. The specimen was placed in the lower assembly of the machine and the force was applied with the help of a knife-like mandrel which engaged the blocks and dislodged it. Bond strength was calculated according to the following formula and expressed in kilonewton:



Fig. 9. The load required to debond the specimen was recorded
 (KN): $\text{Stress} = \text{Failure load (N)}/\text{surface area (mm}^2\text{)}$

2.1 Statistical Analysis

One-way ANOVA was used for data analysis.

2.1.1 Descriptive

Mean value of group A was 4.88, group B 8.83 and group C was 6.63 (P value -0.0675).

3. RESULTS

Bond strength was found to be much higher in group B than in group C and A with a confidence of more than 98.828% (P value -0.0675).

4. DISSCUSION

Acid etching technique using phosphoric acid is well accepted for various applications in dentistry. Among mechanical properties, bond strength of restorative materials is important because it provides sufficient strength to resist intraoral compressive and tensile forces that are produced in function and parafunction [11]. Different etching duration with the same phosphoric acid concentration resulted in different morphologic changes of demineralized dentin surface. A direct correlation between etching time and the depth of demineralized zone was observed. The hybrid layer thickness correlated directly to the etching time [12]. Increased etching time demineralizes dentin surface to a depth greater than that to which resin monomers can penetrate, producing a thick, poorly infiltrated hybrid layer [13].

The recommended acid etching time for dentin with 37% phosphoric acid gel commonly employed with etch and rinse system is 30 seconds.

Ustunkol et al., Batra et al. and Taschner et al. [...,...] claimed that, etching process had a significant effect on bond strength of methacrylate-based composite.

Adebayo et al. showed higher bond strengths of the nano hybrid composite [14].

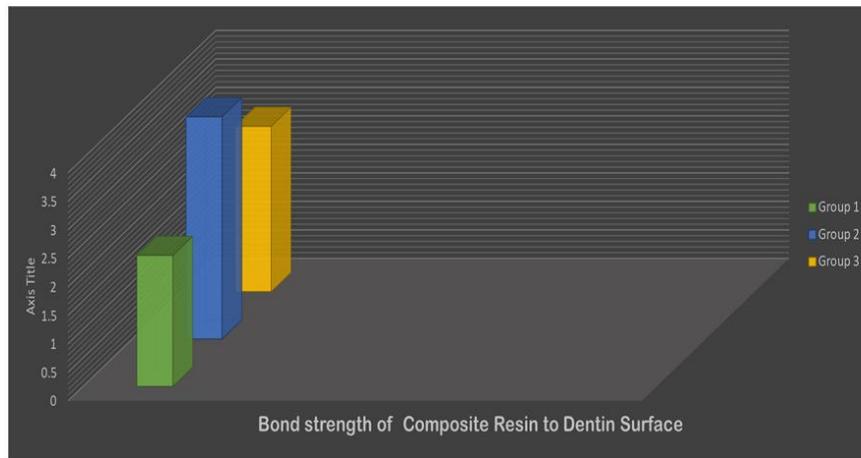


Fig. 10. Bond strength of composite resin with respect to time interval

Table 1.

Groups	Mean deviation	Standard deviation	Sum	Average	Variance
A	4.88	3.15	16.7543	2.347	0.858
B	8.85	0.37	21.664	3.76	1.839
C	6.63	0.73	17.663	2.66	0.676

Table 2.

Source of variation	SS	Df	MS	F	P-value	Fcrit
Between groups	2.33	1	2.4457	21.5674	0.0675	2.5567
Total	2.49	17				

Koliniotou-Koumpia et al. and Sampaio et al. mentioned that, there was no difference of bond strength between nano hybrid composite and bulk fill composite on etching [15]. Dentin is heterogeneous, consisting of hydroxyapatite and collagen. The degree of mineral content in dentin is quite variable, depending on whether it is near the DEJ or deeper in close proximity to the pulp. Acidity of monomer also caused changes in the surface chemistry and morphology of dentin, which in turn can influence the bonding. A significantly thicker hybrid layer was noted in areas with perpendicular tubule orientation than in areas with parallel tubule orientation [16].

Mechanical behaviour depends upon the concentration and particle size of the inorganic filler. For evaluating the bond strength, the study samples were stored in distilled water with few thymol crystals. The purpose was to maintain aseptic conditions before cavity system, with hydrophilic components, which can dislodge moisture from the conditioned dentin. It would also attain an intimate interaction at the demineralized intertubular and peritubular dentin, creating the hybrid layer, which is essential for an ideal bond to dentin which is similar to studies conducted by Kallanos et al. and Gupta et al. [17].

The present study showed that 30 seconds acid etching with 37% phosphoric acid resulted better bond strength than 60 seconds and 15 seconds. Hence, these findings confirmed that the different etching times with the same phosphoric acid concentration resulted in different morphologic changes of demineralized dentin surface. This was very evident in the striking changes in the number, diameter and surface area of dentinal tubules, intertubular surface area, appearance of the dentin surface porous zone containing smear layer and demineralized residual collagen particles with dentin demineralization products in acid globules, and the completely dissolved

peritubular dentin cuff that happened after prolonged etching time [18,19].

5. CONCLUSION

1. When 37% phosphoric acid was applied, a dentin substrate, free of smear layer was not created for the etching time of 15 seconds.
2. Within the limitations of this *in vitro* study; it can be concluded that 30 seconds of etching time gave better bond strength due to longitudinal tubules which influenced the dentin hybridization process in the sense of forming more resin tags that, in turn, contributed to the bond strength.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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