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IN VITRO EFFECT OF ETCHING TIME AND POLYMERIZATION DURATION ON THE SHEAR BOND STRENGTHS TO DENTIN

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Abstract

Introduction: Some authors have used phosphoric acid in different concentrations and etching time for demineralization of tooth hard tissues (Buonocuore, Bertolotti). In this study the *in vitro* effect of dentine etching time with phosphoric acid 37% (20 and 60 seconds) and composite polymerization duration (60 and 180 seconds) on the shear bond strengths to dentin was evaluated.

Materials and methods: The crowns of 32 extracted human molars were cut horizontally with diamond saw to gain larger surface of dentin. The working models were made of auto-polymerizing acrylate with the dentin layer, which was polished. Dentine was treated with 37% phosphoric acid in duration of 20 and 60 seconds, before the fourth generation adhesive (Syntac®) was applied. The control group used the fifth generation adhesive (AdheSe®). The composite was polymerized with two different durations (60 and 180 seconds). The samples stayed in water baths for 24 hours in 37°C. Shear bond strengths were measured with Instron machine. Data were analyzed with Anova and Tuckey test.

Results: Mean values of shear bond strengths for the first group (etching 20 sec / polymerization 60 sec) were 63.8 ± 3.7 N, for the second group (E 60 sec / P 60 sec) 111.7 ± 2.4 N, and for the third group (E 60 sec / P 180 sec) 132.8 ± 14.3 N. The control group (AdheSe®) had mean values 134.4 ± 15.5 N.

Conclusion: There is significant difference found between the second and the first group ($p<0.019$) and between the third and fourth with the first group ($p<0.001$). There is no significant difference between the second, third and fourth group.

Key words: Dentin's shear bond strength, etching time, polymerization.

Introduction

Since there is no direct connection of the composites to the tooth structure, a greatr number of adhesive systems have been developed to accomplish this bond. The foundations of the modern adhesives in dentistry were put in 1955, when Buonocuore reported that acids can be used to modify the enamel's surface and make it "more acceptable for bonding".

Compared to the adhesive systems for enamel, dentin adhesive agents during last twenty years had turbulent history of development. Dentin's heterogeneous composition has made it a more difficult substrate to bond with adhesives than enamel. The smear layer that blocks dentinal tubules acts as a "diffusive barrier". This was initially thought to be beneficial in protecting the pulp decreasing dentin's permeability (Pashley DH et al. 1981). But, since the smear layer is only superficially bonded with the intact dentin's surface, there were poor results for the adhesives applied in this manner (Burke and McCaughey, 1995). The efforts to improve bonding strengths of adhesives to dentin led to introduction of primer solution of the third generation adhesive system in order to change the smear layer. In this generation, eching of dentin partially removed and/or modified the smear layer (Nakabayashi N, Pashley DH, 1998). The complete removal of the smear layer was achieved in the adhesive systems of the fourth generation. The era of today's adhesive systems in the USA has begun by the end of the 1980swhen the total etch concept was introduced, based on the work of Fusayama and others in Japan. Ray Bertolotti and John Kanca in 1991 proposed application of the phosphoric acid to etch enamel and dentin simultaneously. In the fourth generation of the adhesive systems the smear layer is removed completely using organic acids, and is followed by formation of a hybrid layer when primer and adhesive is used (Nakabayashi et al. 1992, 1995), thus achieving stronger bond. The unsuccessful bonding to dentin covered with smear layer before 1990 was due to inability of resins to go through this layer (Tao L, Pashley DH, Boyd L, 1988). In order to alleviate clinical use, one-bottle systems have combined primers and adhesives in one solution, applied after simultaneous enamel and dentin etching (total etch, wet bonding) with 35-37% phosphoric acid for 15-20 seconds (Ferrari M et al. 1997). These systems represent mixture of hydrophilic and hydrophobic resins in diluents, such as acetone, ethanol and water. These adhesive systems crate a mechanism of mechanical bonding of the etched dentin with dentin tags, with lateral adhesive branches, and thus creating a hybrid layer

which showed higher values of shear bond strengths to enamel and dentin (Tay FR et al. 1994). Watanabe and Nakabayashi in 1993 have developed a self-etching primer, which is water solution of 20% phenyl-P in 30% HEMA, simultaneously bonding both dentin and enamel.

The combination of steps of etching and priming reduces working time, eliminates the rising of the acids` gel and avoids the collagen collapse. Bot the self-etching primer has some cons, e.g. the solution has to be refreshed continuously, because the composition of the solution cannot be controlled (Ferrari et al. 1997) and often here is still smear layer left between the adhesive and dentin (Nakabayashi et al. 1998).

Toida et al. (1995) advised that the removal of the smear layer with a separate step of etching before the bonding would produce stronger and sustainable bond to dentin. Tests of the shear bond strengths *in vitro* often didn`t show significant differences between one-bottle systems and adhesives with sel-etching primers. *In* vitro and clinical microleackage tests showed that one-bottle systems produced better sealing with dentin margins than self-etching systems (Ferrari M, Manocci F, 1997).

Al-Ehaideb et al. (2000) studied shear bond strengths to dentin of five one-bottle self-etching systems. Blomlof et al. (2001) compared action of 32% of phosphoric acid in 15 seconds and EDTA 24% in 3 minutes. Cederlund et al. (2001) treated dentin with EDTA 24% in 30, 60, 120 and 240 seconds. Abu-Hanna and Gordan (2004) evaluated the impact of etching time with phosphoric acid (5, 15 and 30 seconds) in bonding strengths to dentin. Erhardt et al (2004) used phosphoric 37% in 15 seconds.

Lately, adhesive systems have developed further, and a sixth generation is proposed. Those systems have the possibility of achieving a proper bonding to enamel and dentinusing only one solution. The materials of this generation should be used in a single step. Unfortunately, initial evaluations of these new systems showed sufficient bonding to dentin, but had less effective bonding to enamel. This is due to acidic content of the sixth generation adhesive systems.

According to data on polymerization of the composites, until now, it has been proved that the thickness of composites and the duration of polymerization have impact on the composites` solidity. No doubt that this is one of key factors of composite bonding to dentin (Sustaric et al. 1994). According to this investigation, it is confirmed that the solidity of

the composites between the top and bottom layer (surface in contact with dentin) is not the same.

The aim of this study was to test the *in vitro* shear bond strength of composites to dentin after etching with phosphoric acid 37% with 20 and 60 seconds durations and with different polymerization durations (60 and 180 seconds).

Material and methods

For this study were used 32 fresh intact molars extracted for orthodontic purposes, with complete crown. The teeth were immersed in sterile saline, washed and cleane with brush and paste. The crowns were cut horizontally using Isomet 1000 Precision Saw (Buegler Ltd) in the "Joze Stefan" Institute in Ljubljana, Slovenia.

The prepared tooth faced a quartz plate with its dentin surface and around was placed a quartz cylinder (O 15). The cylinder was filled with autopolymerizing resin (Probase Cold, Ivoclar, Lichenstein). In order to have precise results, the dentin surface was planned and polished using polishing machine (Pendex-2, Denmark) from the same Institute. Samples were prepared in the scientific-research laboratory of the Dentistry School of University of Ljubljana, Slovenia.

The samples were randomly placed in four groups of eight.

The first three groups were used to test the adhesion of the Syntac system depending on the dentin etching time and polymerization duration of the composite, and the fourth group was used as a control using AdheSe system.

In the first group dentin was etched in 20 seconds and polymerization duration 60 seconds. The second group had 60 seconds of etching and same polymerization time as the first group. The third group had same etching time as the second group and polymerization 180 seconds. The fourth group used fifth generation of adhesive system (AdheSe) with polymerization of the composite of 60 seconds. The composite used in all groups was Tetric Ceram (Vivadent, Lichtenstein).

After the removal of the plastic ring used during polymerization, the workin mode was ready to be tested for shear bond strength. The models were immersed in distilled water in

37C for 24 hours. The composites bonding to dentin was determined using the test of shear bond strength based on the standard ISO TR 11405/1994.

Shear bond strength was tested using Instron Testing Maching (Instron 4301) in "Joze Stefan" Institute, in Ljubjana, Slovenia. The results were expressed in Newton (N).

The statistical analysis were done using one way analysis of variance (ANOVA) and Tuckey test.

Results

The testing of the shear bond strengths of the composite to dentin showed the following results:

- The first group had mean bond strength 63.8 +- 3.7 N
- The second group the strength was significantly higher with mean 111.7 +- 2.4 N\
- The third group had mean strength 132.8 +- 14.3 N.
- The fourth group had the highest mean shear bond strength – 134.4 +- 15.5 N.\

The one way analysis of variance, used to compare the mean vaues between the tested groups and resulted with significant statistical difference ($p<0.001$).

Source of Variation	DF	SS	MS	F	P
Between groups	3	25995.6	8665.2	9.4	<0.001
Residual	28	25934.5	926.2		
Total	31	51930.1			

In order to determine the significant difference between groups, multiple comparison with Tukey test was used. From this analysis we fond significant difference between the second and the first group ($p<0.019$) and between groups three and four with the first group ($p<0.001$, respectively).

No significant difference was found between the third and second, fourth and second and fourth and third group.

Comparison	Diff of means	p	q	Significance P
Gr II vs Gr I	47.93	4	4.45	< 0.019

Gr III vs Gr I	69.00	4	6.41	< 0.001
Gr IV vs Gr I	70.57	4	5.56	< 0.001
Gr III vs Gr II	21.08	4	1.96	> 0.519
Gr IV vs Gr II	22.65	4	2.11	> 0.458
Gr IV vs Gr III	1.57	4	0.15	> 1.0

Conclusion

Based on the results, we can conclude that with longer etching time of dentin with phosphoric acid 37%, as well as longer duration of composite polymerization the shear bond strengths of composite with dentin is increased.

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