European Journal of Research in Dentistry 2020; 4-1: 12-17
DOI: 10.35333/ERD.2020.181

ORIGINAL ARTICLE / ÖZGÜN ARAŞTIRMA

Effects of Different Solvents on the Shear Bond Strength of A Nano Additive Composite

Farklı Çözücü İçerikli Adezivlerin Nano Doldurucu Katkılı Kompozitlerin Makaslama Bağlanma Değerleri Üzerine Etkisi

Gökhan DOKUMACIGİL, Pınar YILMAZ ATALI, Faik Bülent TOPBAŞI

Abstract

Aim: This study evaluated the effect of different solvents on shear bond strength (SBS) and how they were affected by the aging process after 10,000 thermocycling.

Materials and methods: Forty intact human molars were randomly divided into 4 groups of 10 teeth each. The teeth were embedded into self-curing acrylic resin and the occlusal enamel of teeth were cut with a low-speed diamond saw (Isomet 2000, USA). Two self-etching bonding agents containing different solvents, All-in-One (OptiBond, KavoKerr, USA), N-Bond (Tetric, Ivoclar Vivadent, Liechtenstein) were applied over the mid-dentin surface following the manufacturers' instructions. Subsequently, two-millimeter columns of Tetric N-Ceram (Ivoclar Vivadent, Liechtenstein) composite (Shade A2) were placed over the adhesives. Then the specimens were subjected to SBS test by using a universal shear testing machine. While two groups were tested after 24 h following the polymerization, the other groups were tested after 10,000 times thermal aging. The types of failure were evaluated under 25X magnification. The significance level $p<0.05$ was used for all hypothesis tests.

Result: The One-way ANOVA revealed no significant difference in SBS values between OptiBond All-in-one and Tetric N-bond in the different experimental conditions, immediately (p=0,585) and after 10,000 thermocycling (p=0.266). But all thermocycling groups showed lower bond strength than immediate groups (p<0,05). Besides, the types of failure were affected by thermal aging.

Conclusion: The SBS values were not affected by the solvent type. However, the thermal aging reduced the SBS values for both adhesive agents.

Keywords: Adhesive, Solvent, Composite, Shear Bond Strength

Gökhan DOKUMACIGİL(✉)
Marmara University, Faculty Of Dentistry, Department of Restorative Dentistry, Istanbul, Turkey
e-mail: gdokumacigil@gmail.com

Pınar YILMAZ ATALI
Marmara University, Faculty Of Dentistry, Department of Restorative Dentistry, Istanbul, Turkey

Faik Bülent TOPBAŞI
Marmara University, Faculty Of Dentistry, Department of Restorative Dentistry, Istanbul, Turkey

Submitted / Gönderilme: 17.01.2020 **Accepted / Kabul:**28.06.2020

Öz

Amaç: Bu çalışmanın amacı farklı çözücüler içeren dental adezivlerin immediyat ve 10.000 devir ısısal döngü uygulamasından sonra dentine olan makaslama bağlanma dayanımlarının karşılaştırılmasıdır.

Gereç ve Yöntem: Çürüksüz ve restorasyonsuz 40 adet insan azı dişi her grupta 10 tane diş olacak şekilde rastgele 4 gruba ayrılmıştır. Dişler soğuk akrilik içerisine gömüldükten sonra su soğutması altında kesme cihazıyla (Isomet 2000, ABD) oklüzal mine ortadan kaldırılmıştır. Farklı çözücüler içeren 2 adet kendinden asitli (*self-etch*) adeziv, All-in-one (Optibond, KavoKerr, ABD), N-Bond (Tetric, Ivoclar Vivadent, Lihtenştayn), üreticilerin talimataları doğrultusunda dentin yüzeyine uygulanmıştır. Daha sonra adezivlerin üzerine iki milimetre kalınlık ve 4 milimetre çapında A2 renk Tetric N-Ceram (Ivoclar Vivadent, Lihtenştayn) kompozit blokları oluşturulmuştur. Numuneler hazırlandıktan sonra universal test cihazı ile (Trapezum X, Shimadzu, Japonya) makaslama kuvvetine maruz bırakılmıştır. Oluşturulan 4 gruptan 2 tanesi polimerizasyon aşamasını takip eden 24 saat sonra test edilirken, diğer 2 grup 10.000 devir ısısal döngü uygulaması sonrası test edilmiştir. Kopma türleri 25X büyütme altında değerlendirilmiş ve tüm hipotez testlerinde istatistiksel anlamlılık değerleri $p<0.05$ olarak değerlendirilmiştir.

Sonuç: Tek yönlü varyans analizine (ANOVA) göre yapılan değerlendirmelerde Optibond All-in-one ve Tetric N-bond adezivin makaslama bağlanma değerleri arasında farklı ortam koşullarında, immediyat (p=0,585) ve 10.000 devir ısısal döngü uygulaması sonrasında (p=0.266), anlamlı bir fark bulunmamıştır. Fakat ısısal döngü uygulanan gruplar immediyat gruplara göre anlamlı derecede düşük bağlanma değerleri göstermiştir. ($p<0.05$) Ayrıca kopma tipleri ısısal döngüden etkilenmiştir.

Sonuç: Bu çalışmanın ışığında, makaslama bağlanma değerleri çözücü tiplerinden etkilenmezken, ısısal döngü uygulaması makaslama bağlanma değerlerini anlamlı derecede düşürmüştür.

Anahtar kelimeler: Adeziv, Çözücü, Kompozit, Makaslama bağlanma dayanımı

INTRODUCTION

Today, resin restorations are widely used in dentistry. One of the most critical steps of these restorations is adhesion that

ensures mechanical and physical forces between different materials by using various components. The adhesive interface consists of a substrate (adherent) and "adhesive", which placed on the substrate (2). The adhesive systems are used for bonding resins to enamel and dentin. The ultimate aim of bonding agents is to achieve maximum adaptation of the dental substrate with the restorative material.

Current developments in dentistry are based on reduced application time and simplification. Therefore, self-etch adhesive systems are widely used in dentistry. Because, these techniques do not require an additional acid-etch step and they are simultaneously dissolving the smear layer partially as they are infiltrating to dentin tubules (3). The first self-etch adhesives were consisting of two bottles, a bonding resin, and an acidic primer. Recently, many clinicians prefer to use one-step self-etch adhesives called as all-in-one (AIO) systems in which all the components have been combined into a single bottle. All-in-one systems require fewer steps for the bonding procedure, so they are easy to use because of reducing the application time.

Notwithstanding the dental bonding agents ensure the adhesion, shrinkage of the dentin matrix is another problem. To solve this problem, different solvents have been added into dental adhesives such as water, ethanol, and acetone. Water is one of the most essential solvents as it contributes to the ionization of acidic monomers (3). Also, it surrounds the residual collagens, which avoid hydrogen bonding. This preserves the inter-fibrillary spaces, thus resin materials could infiltrate to these spaces (4). Ethanol is a different solvent, which evaporates better than water because of higher vapor pressure. Generally, water is co-solvent of ethanol containing adhesives (5). The other important solvent is acetone and it has higher water removing capability than ethanol (6). Due to its high volatility, acetone-containing adhesives has a lower shelf life than the others (7). Consequently, the clinician must pay attention to storage conditions for this kind of bonding systems.

Having said that, durability of resin materials may decrease in time. The widely accepted method is thermocycling to simulate the aging in resin bonds (8). Especially, the restorations performed using self-etch systems may be affected more than the etch and rinse systems after thermocycling due to their hydrophilic ingredients.

This study evaluated the shear bond strength (SBS) values of 3 solvent containing adhesive by comparing ethanol/water-containing adhesive as immediate and after 10,000 thermocycling. The null hypothesis was that SBS to dentin is unaffected by solvent type, either immediately or after thermocycling.

MATERIALS AND METHODS

Marmara University Scientific Research Projects Commission (BAPKO) supported this study (Project no: SAG-C-DUP-120.917.0507). This study is approved by the ethical committee of Marmara University Faculty of Dentistry with the ethical number 2017-84 dated on 27.03.2017.

Study Design

Extracted human molar teeth within the previous 6 months were collected, residual tissue were removed, stored in 0.1% thymol solution, and finally they were embedded acrylic resin. The occlusal enamel of teeth was cut by low-speed diamond saw (Isomet 2000, Buehler, USA). To simulate the clinical dentin condition, surfaces were roughened by using mid-grain diamond burs (125 μm). The teeth were divided into 4 groups of 10 teeth (Group I (a, b), Group II (a, b). Two self-etching bonding agents, All-in-One (OptiBond, KavoKerr, USA) containing acetone/ethanol/water and N-Bond (Tetric, Ivoclar Vivadent, Liechtenstein) containing only ethanol/water, were applied for 40 and 10 s, respectively. Following the application of the bonding agents on the dentin surface, they were thinned with a gentle stream of air and cured for 10 s using a LED curing unit (VALO Cordless, Ultradent, USA) with an output of 1000mW/cm^2, as suggested by the manufacturer. The protocols for application and composition of the materials used in the study have been shown in Table 1. Two-millimeter columns of Tetric N-Ceram (Ivoclar Vivadent, Liechtenstein) composite with the shade A2 were placed over the mid-dentin surface by using silicone molds (2 mm thickness and 4 mm diameter) and a mylar strip to attain flat-ended surfaces, and cured for 10 s with the same visible light curing unit. Subsequently, during the control groups (Group Ia and Group IIa) were stored in 37 °C distilled water for 24 h, the test groups (Group Ib and Group IIb) were subjected to 10,000 thermocylcing between two water baths, a cold water bath at 5 °C and a warm water bath 55 °C.

Group I: Optibond All-in-one(AIO) + Tetric N-Ceram (TNC)

a) Immediately

b) After 10,000 thermal cycles

Group II: Tetric N-Bond Universal + Tetric N-ceram (TNC)

a) Immediately

b) After 10,000 thermal cycles

Afterwards, the samples were placed in a universal testing machine (Trapezium X, Shimadzu Corporation, Japan) and tested in shear to failure with a 0.5 mm/min crosshead speed. SBS values were calculated as MPa units.

Table 1. The protocols for application and composition of the materials

Brand	LOT	Composition	Procedures
Optibond All-in-one (KavoKerr, USA)	**33381E**	GPDM, mono and di-functional methacrylate monomers, nano-fillers including sodium hexafluorosilicate, water, acetone and ethyl alcohol, photoinitiators ph: 2.5	Two coats, Apply for 20 s, For each coat, dry gently, light cure for 10 s with 1000 mW/cm^2 power
Tetric N-Bond Universal (Ivoclar Vivadent, Liechtenstein)	**W86807**	Methacrylate, water, ethanol, silicon dioxide, photo stabilizers, stabilizers ph: 2.5 – 3.0	Scrub for 10 s, dry gently, light cure for 10 s with 1000 mW/cm^2 power
Tetric N-Ceram (Ivoclar Vivadent, Liechtenstein)	**W84900**	Barium glass, yitterbium triflouride, silicon dioxide **Matrix:** Bis-GMA, TEGDMA, ethoxylated Bis-EMA, UDMA	10 s. Light cure with 1000 mW/cm^2 power

Statistical analysis

The mean SBS to dentin was compared for 2 materials (Optibond All-in-one and Tetric N-bond) under 2 conditions; immediately and after thermocycling (10,000) using a one-way analysis of variance (ANOVA) and Tukey's tests at a significance level of 0.05. The statistical software package (NCSS, Utah, USA) was used to perform statistics.

Failure analysis

The types of failures were determined under 25X magnification (Leica, USA) and recorded as either "adhesive failure", "cohesive failure within dentin", "cohesive failure within composite" or "mixed failure".

RESULTS

The Mean SBS values and standard deviations (SD) for different groups are presented in Table 2. Bonding to dentin surface with Tetric N-bond resulted in higher bond strengths in both immediate (15.47±2.78 MPa) and thermocycled groups (11.06±2.67 MPa) than Optibond All-in-one (immediate: 14.56±4.3 MPa; thermocycled: 9.75±2.43 MPa). However, the One-way ANOVA revealed no significant difference in SBS values between OptiBond All-in-one and Tetric N-bond in the immediate (p=0.585) and thermocycled groups (p=0.266). The groups subjected to thermal aging showed lower SBS values compared to immediate groups (p=0.02 for OptiBond All-in-one; p=0.017 for Tetric N-bond) For all groups, most of the bonding failures were observed as either adhesive fracture or mixed fracture showing some attached materials or fractures on the dentin surface. However, there was no cohesive failure of the resin in any of the groups (Table 3). Thermocycling induced the morphological changes of failure type and increased the adhesive failure rate of the groups. Although the Optibond All-in-one group showed more adhesive failure and less cohesive failure within dentin after thermocycling compared to immediate rates, the failure types of Tetric N-bond has not been affected by thermocycling as much as AIO.

Table 2. Mean SBS values and Standard Deviations (SD)

Composite	Adhesive	Immediate SBS	SBS after Thermocycle	p
Tetric N-Ceram	**OptiBond AIO**	14.56 ± 4.37	9.75 ± 2.43	0.02
	Tetric N-Bond Universal	15.47 ± 2.78	11.06 ± 2.67	0.017
p<0.05				

Table 3. Failure modes

Testing Procedure	Composite	Adhesive	Cohesive Failure (Resin)	Cohesive Failure (Dentin)	Adhesive Failure	Mixed
Immediate	Tetric N-Ceram	Optibond AIO	-	3	2	5
		Tetric N-Bond Universal	-	1	3	6
10,000 Ther mocycle	Tetric N-Ceram	Optibond AIO	-	1	5	4
		Tetric N-Bond Universal	-	2	3	5

DISCUSSION

One of the most critical steps of the resin restoration technique is adhesion. Conversely to the former procedures which clinicians had to drill not only infected tooth tissue but also healthy tissues to enhance the mechanical retention (2), adhesive systems supply restoring teeth with minimally invasive procedures using various materials.

Etch and rinse systems increase the SBS, but self-etch adhesives have been improved and simplified while providing better durability and they were introduced as an alternative because of their reduced application time and sensitivity of the technique (9).

Faye et al. compared SBS of different self-etch adhesives by prior etching and non-etching. As a result, they found significantly higher SBS in prior etching groups (10).

Despite the limited research data, the available studies show that one-step AIO systems provide better long-term retention. Kubo et al. (11) evaluated the SBS of 2 different self-etch adhesives and found a retention rate of 98.1% in two-year follow-up. Apart from this, using the etch and rinse systems, extremely drying of dentin surface may lead to the structural collapse of the collagen matrix, which results in inadequate infiltration of monomers into the matrix. The prevention of the teeth from this collapse is made possible by providing adequate moisture within dentin structure (2). The organic solvents, such as acetone and ethanol, which improve monomer infiltration into the demineralized dentin matrix, play a crucial role to remove excessive water during evaporation and dissolve the resin monomers (12).

Up-to-date materials are introduced in restorative dentistry. Although there are several resin composites available in the market, the recently introduced resin composites have been produced by nano-filler technology which is one of the most important reasons of the improvement in the dental composites. Nayak et al. compared the SBS of 4 new nano-filled composites with the orthodontic light cure resin Transbond XT (3M ESPE, USA) and reported that Tetric N-ceram composite and the orthodontic resin showed higher SBS values (13).

Two of the important factors to attain a high quality hybrid layer are optimal monomer infiltration into the collagen fibrils of the demineralized dentin matrix and the removal of excessive water and organic solvents from the surface prior to curing (1). There is still controversy over which is the better solvent (acetone or ethanol). Acetone is widely used as a solvent, due to its efficiently water removal capability from the surfaces. However, acetone-containing adhesives has lower shelf life because of their high volatility. Ethanol is another material used as an organic solvent in the adhesives, but its vapor pressure is lower than acetone. In consequence of its more hydrogen bonding capacity, water has not chased by alcohol as effective as acetone (7). Conversely, ethanol can expand dried demineralized matrix more than acetone. Therefore, acetone-containing adhesives must be used and preferred only with the wet-bonding technique (14). Nevertheless, "dry-bonding" technique, namely gentle drying after rinsing is still providing effective adhesion for the water/ethanol-based adhesives (15).

In a study concerning the effect of solvent type on bond strength, ethanol-based adhesives resulted in higher bond strength values than acetone-based adhesives (16). The controversial results regarding the effect of solvent type on bond strength might be on the ground of both of the adhesives contain ethanol solvent.

In a study by Atash and Van den Abbeele, it has been suggested that the bond strength of adhesives depends on the pH value (17). The pH values of dentin adhesive systems used in our study were similar, and they were 2.5 for the OptiBond All-in-one system whilst between 2.5-3 for the Tetric N-Bond Universal system.

It was stated that adhesives including different solvents may show different bond strength values in superficial and deep dentin according to dentin bonding protocols (18). It is much more difficult to achieve adhesion in deep dentin than superficial dentin due to decreased intertubular dentin area in the deep dentin and the increased water content (19).

The SBS to the superficial dentin was regarded as higher independently from the adhesive system. In this study, the

mid-dentin surfaces of the teeth were used for the application of adhesive and resin composite (20).

Furthermore, there are lots of studies about SBS after 24-h storage, but some researchers have evaluated SBS values after thermocycling. The *in vitro* aging methods are often preferred as an alternative method to *in vivo* aging studies. It also has been reported that 10,000 thermal cycles have a similar effect with 1 year of aging in the mouth (21). The main factor that negatively affects the adhesive surface is the hydrolysis of resin-dentin bonding. Hot water may resolve poorly polymerized resin monomers and increase hydrolysis of collagen structure (1). In this study, both groups have been affected by thermocycling and this decreased the SBS values.

According to the literature, it was seen that the SBS tests were preferred more than the tensile bond strength tests (22) by reason of the fact that shear forces mimic the clinical conditions better than tensile forces (23). According to these results, the SBS comparison was chosen for this study.

Failure modes were subdivided as cohesive, adhesive, cohesive in dentin, and cohesive in resin. It has been reported that SBS plays an important role in failure types. Although adhesive failure occurs in the lower SBS values, cohesive failures occur in higher SBS values (24). The adhesive failures might have a couple of reasons such as inadequate wetting, excessive water or excessive air-drying (25). Although this study has given information about the effects of solvent type on SBS, this *in vitro* study does not fully reflect the real conditions of the oral cavity.

CONCLUSION

Under the limitations of this *in vitro* study, it can be concluded that;

1. There was no significant difference between the bond strength values of ethanol/water containing and acetone/ethanol/water containing adhesives.
2. The thermal aging has reduced the bond strength of all tested materials.

More information is needed about laboratory and clinical performance of the three-solvent containing adhesives.

REFERENCES

3. Hashimoto M, Fujita S, Nagano F, Ohno H, Endo K: Ten-years degradation of resin-dentin bonds. Eur J Oral Sci 2010;118:404-410
4. Ozcan M, Dundar M, Comlekoglu ME. Adhesion concepts in dentistry: tooth and material aspects. J Adhes Sci Technol 2012;26:2661-2681
5. Perdigao J. New developments in dental adhesion. Dent Clin North Am 2007;51:333-357
6. Tay FR, Pashley DH. Biomimetic remineralization of resin-bonded acid-etched dentin. J Dent Res 2009;88:719-724
7. Moszner N, Salz U, Zimmermann J. Chemical aspects of self-etching enamel-dentin adhesives: a systematic review. Dent Mater 2005;21:895-910
8. Lee SB, Cabezas CG, Kim KM, Kim KN, Kuroda K. Catechol-functionalized synthetic polymer as a dental adhesive to contaminated dentin surface for a composite restoration. Biomacromolecules 2015;16:2265-2275
9. Van Landuyt KL, Mine A, De Munck J, Countinho E, Peumans M, Jaezcques S, Lambrechts P, Van Meerbeek B. Technique sensitivity of water-free one-step adhesives. Dent Mater 2008;24:1258-1267
10. Erickson RL, Barkmeier WW, Kimmes NS. Bond strength of self-etch adhesives to pre – etched enamel. Dent Mater 2009;25:1187-1194
11. Peumans M, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Five-year clinical effectiveness of a two-step self-etching adhesive. J Adhes Dent 2007;9;7-10
12. Faye B, Sarr M, Bane K, Aidara AW, Niang SO, Kane AW. One-year clinical evaluation of the bonding effectiveness of a one-step, self-etch adhesive in noncarious cervical lesion therapy. Int J Dent 2015;2015:984065
13. Kubo S, Yokota H, Hayashi Y. Two-year clinical evaluation of one-step self-etch systems in non-carious cervical lesions. J Dent 2009;37:49-155
14. Reis AF, Oliveira MT, Giannini M, De Goes MF, Rueggeberg FA. The effect of organic solvents on one-bottle adhesives' bond strength to enamel and dentin. Oper Dent 2003;28:700-706
15. Nayak RS, Valecha S, Pasha A, Mamatha J, Khanna B, Shafiuddin B. A comparison of shear bond strength of new nanofilled composite and nano-ionomer restorative materials with traditional adhesive material for orthodontic bracket bonding: an in vitro study. J Int Oral Health 2015;7:70-76
16. Pashley DH, Agee KA, Carvalho RM, Lee WK, Tay FR, Callison TE. Effects of water-free polar solvents on the tensile properties of demineralized dentin. Dent Mater 2003;19:347-352
17. Manso AP, Marquesina Jr L, Silva SMA, Pashley DH, Tay FR, Carvalho RM. Stability of wet versus dry bonding with different solvent-based adhesives. Dent Mater 2008;24:476-482
18. Cardoso PC, Lopes GC, Vieira LCC, Baratieri LN. Effect of solvent type on microtensile bond strength of a total-etch one-bottle adhesive system to moist or dry dentin. Oper Dent 2005;30;376-381
19. Atash R, Van den Abbeele A. Bond strengths of eight contemporary adhesives to enamel and to dentin: an in vitro study on bovine primary teeth. Int J Pediatr Dent 2005;15:264-273

20. Toledano M, Osorio R, Ceballos L, Fuentes MV, Fernandes C, Tay FR, Carvalho RM. Microtensile bond strength of several adhesive systems to different dentin depths. Am J Dent 2003;16:292-298
21. Giannini, M, Carvalho RM, Martins L, Dias C, Pashley DH. The influence of tubule density and area of solid dentin on bond strength of two adhesive systems to dentin. J Adhes Dent 2001;3:315-324
22. Pegado RE, Do Amaral FL, Florio FM, Basting RT. Effect of different bonding strategies on adhesion to deep and superficial permanent dentin. Eur J Dent 2010;4:110-117
23. Bektas OO, Eren D, Siso SH, Akin GE. Effect of thermocycling on the bond strength of composite resin to bur and laser treated composite resin. Lasers Med Sci 2012;27:723-728
24. Nakajima M, Sano H, Burrow M, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell C, Pashley DH. Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesives. J Dent Res 1995;74:1679-1688
25. Cardoso PE, Braga RR, Carrilho MR. Evaluation of micro-tensile, shear and tensile tests determining the bond strength of three adhesive systems. Dent Mater 1998;14:394-398
26. Dundar M, Ozcan M, Gökçe B, Comlekoglu E, Leite F, Valandro LF. Comparison of two bond strength testing methodologies for bilayered all-ceramics. Dent Mater 2007;23:630-636
27. Spreafice D, Semeraro S, Mezzanzanica D, Re D, Gagliani M, Tanaka T, Sano H, Sidhu SK. The effect of the air-blowing step on the technique sensitivity of four different adhesive systems. J Dent 2006;34:237-244