Comparison of Microtensile Bond Strength of Silorane-Based Composite with the Conventional Methacrylate Composite to the Dentin of Primary Teeth

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Abstract

Statement of Problem: The bond strength between restorative material and tooth structure is so important for conduction of durable restoration. Considering the recent attention to low shrinkage composite resins, evaluation of micro tensile bond strength of these materials would be valuable.

Objectives: To compare the microtensile bond strength of silorane composite resin (Filtek P90) with the conventional methacrylate composite (Filtek Z250) with and without applying acid etch before application of bonding system.

Materials and Methods: In this experimental study, 24 intact primary canines were used. After the dentin was exposed, the teeth were randomly divided into four groups as follows: the first group (silorane bond system + composite Filtek P90); the second group (etch + silorane bond system + composite Filtek P90); the third group (Single bond + composite Filtek Z250); and the fourth group (etch + Single bond + composite Filtek Z250). The teeth were cut on the longitudinal axis and the interface between the composite and dentin were grinded buccolingually and mesiodistally. The samples were subjected to a microtensile force until breakage. The obtained values were recorded in MPa; data were analyzed using One-way ANOVA and Tamhane’s T2 statistical tests.

Results: The average microtensile bond strength in all groups had a statistically significant difference with each other (all p < 0.05). The highest bond strength belonged to the second group (etch + silorane bond system + composite Filtek P90) and the lowest value was related to the third group (Single bond + composite Filtek Z250).

Conclusions: As the second group (etch + silorane bond system + composite Filtek P90) exhibited higher microtensile bond strength, it may prove that using composite Filtek P90 is preferable to be used in primary dentin in comparison with composite Filtek Z250, and using etch + silorane bond system is more advantageous than single bond system.

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Introduction

Extensive research continues to create a better restorative material and the more advanced and applicable techniques in the restorative dentistry. The highest recent efforts in response to the growing needs and expectations are the usage of composite resin. The resin composite restorations have been accepted due to their great beauty and having other benefits such as low heat transfer ability, and tooth structure protection during cavity preparation [1,2]. Physical properties of composite dental material have been improved during decades, but bond strength, specially to the dentin, has still remained a challenge [3,4].

If the bond between the restoration and tooth fails, recurrence caries occurs and higher destruction of dental tissue is seen [5]. Micro-leakage poses a particular problem for the teeth in the paediatric patient because the floor of the cavity preparation may be close to the pulp. Added insult to the pulp is caused by the seepage of the irritants that penetrate around the restoration and through the thin layer of dentin, or a microscopic pulpal exposure may produce irreversible pulp damage [2].

The volumetric shrinkage for traditional methacrylate-based composites ranges from 2% to 6%. Similarly, the marginal gap rate in the clinical assessment of the resin composite restorations based on the material type and clinical application method has a range of 14% to 54% [5]. The volumetric shrinkage of the composite resins is mainly dependent on the chemical composition of the resin matrix. Therefore, the efforts have been made to reduce the polymerization shrinkage with the change in the resin composition, including strengthening of the conventional composite resins Bis-GMA via the addition of the new monomers such as the UEDMA and Bis-EMA and or utilization of the longer resin molecules such as EMA6 in Filtek Z250 [6]. The other facts of these efforts are to change the monomeric phase matrix structure with the use of the ring-opening monomers [7].

The conducted researches [8,9] have shown that the reinforced formula with Oxirane/Polyol had a determined reduction in the polymerization shrinkage and cusp deflection in relation to the conventional methacrylate composite resins. The polymerization contraction reduction was in association with the cationic-ring-opening monomers. Concerns about cytotoxicity and mutagenicity of the resins with the Oxirane/Polyol base existed in the clinical application. In order to solve the raised problems, a new resin formula was obtained from the Oxirane and Silorane molecule reaction that was entitled ‘Silorane’ [8].

The silorane had very good tissue compatibility and its cytotoxicity is equivalent or less than the conventional methacrylate monomers. Silorane is insoluble and stable in the biological solutions. Even in comparison to the composite resins with methacrylate base, it causes less cusp deflection. In the conducted studies, it has been shown that silorane possesses optimal mechanical characteristics as a restorative material. The ring-opening reaction present in the silorane has reduced the polymerization shrinkage volumetric rate to less than 1% [8-10].

In the recent years, numerous researches have studied the characteristics of this new resin composite and its clinical application; many of these researches have focused on the application of this kind of resin composite on the permanent teeth [10-12]. Yet, sufficient laboratory or clinical studies showing usefulness of this new resin composite in the primary teeth have not been conducted. In the primary teeth, in contrast to the permanent teeth, the thickness of the dental material is less and that of the prismless enamel is high; therefore, the results of the resin composite application and the bond system in the permanent teeth cannot be generalized to the primary teeth [11].

Contemporary dental adhesive systems can be classified according to application techniques as etch and rinse or self-etching adhesive systems. Manufacturers combine together either primer and bonding resin or etchant and primer agent to reduce the number of bottles and clinical steps. Also they simplify the bonding protocol by producing all-in-one systems. An important indicator of an adhesive systems’ effectiveness is bond strength; Because the bonding layer must support composite shrinkage stress and occlusal forces to avoid gap formation which leads to micro-leakage, secondary caries, and postoperative sensitivity [9,13].

Therefore, there is a need to conduct a study assessing the silorane application to the primary teeth dentin is felt. Numerous methods are used for evaluating the bond strength rate of the restorative materials and bonding system, among which the shear and tensile bond test can be addressed [14].

For the measurement of the tensile bond strength, recently the Micro Tensile Bond Strength (μTBS) technique has been introduced via Sano [15]. This new method has many advantages over standard
methods for measuring the bond strength; they include:
1- It provides the possibility to prepare several resin cylinders joint to the dentin in a dental sample. 2- It provides the possibility to test the dental levels with different clinical characteristics such as decayed dentin, dentinal tubule sclerosis and enamel surfaces. 3- With the reduction of experiment surface, the superficial defects and distinctiveness reduces and causes prevention of inappropriate distribution of force and premature bond collapse. 4- It provides the possibility for testing the localized differences in the bond strength in the tooth [7,8].

The present study was conducted aiming at comparing micro- tensile bond strength of the silorane composite Filtek P90 with the conventional methacrylate composite Filtek Z250 to the primary teeth dentin.

**Material and Methods**

In this experimental study, 24 primary canines without decay, fracture, and cracks which were extracted due to orthodontics treatment were used. For teeth disinfection, after cleaning of the tissue wastes with scaler, they were placed in 10% formaldehyde solution (Dr. Mojalali, Tehran, Iran) for a period of 24 hours. Using diamond grinding fissure bur number 012 (Tizkavan, Tehran, Iran) we removed the coronal part of the canine teeth and exposed the dentin surface. Then, the surfaces were investigated under steriomicroscop for pulp horns or microscopic pulp exposure. If there was no microscopic pulp exposure, the teeth were randomly divided into the four groups (n = 6). The prepared teeth surface was washed with water and air aspirator for a period of 15 seconds and dried and after that in each group the dentin surface was covered with the following composite, respectively. The composite with an incremental technique was placed in the two layers of two millimetres [16,17].

*Group A: Composite Filtek P90 with an individual self etch bond system*

In this group, according to the manufacture’s instructions the primer existing in the special composite kit; Silorane Filtek P90 (3M ESPE, MI, USA) was ‘brushed’ on the sample surface for a period of 15 seconds and diffused for five seconds with a gentle air aspiration; then, it was cured for a period of 10 seconds via light curing machine QTH (Coltolux 75, OH, USA) with 1000mw/cm² light intensity. Later the special composite bonding Filtek P90 was applied to the samples’ surface and after the diffusion with the gentle air aspiration was cured for a period of 10 seconds. Eventually, the composite Filtek P90 was conformed in two layers of two millimeters on the dentin surface and each layer was cured for a period of 40 seconds.

*Group B: Composite Filtek P90 with the use of acid etching and an individual bond*

In this group, the work stages were similar to Group A with a difference that before the primer use, the dentin surface was etched with the use of 37% phosphoric acid (3M ESPE, MI, USA) for 15 seconds, washed for a period of 10 seconds and dried.

*Group C: Composite Filtek Z250 with the self etch bond system:*

In this group, the self etch bonding system, Single bond, as per the manufacturing company’s instruction was used for bonding. Then, the composite Filtek Z250 similar to the earlier groups was conformed to the dentin surface and cured for a period of 40 seconds.

*Group D: Composite Filtek Z250 with the use of acid etching and self-etch bond system:*

In this group, the dentin surface was etched for 15 seconds with the use of 37% phosphoric acid and then washed for a period of 10 seconds and dried. Then, the self etch bonding system, Single bond, (3M ESPE, MI, USA) was used for bonding based on the manufacturer’s instruction. Then, the composite Filtek Z250 similar to the earlier groups was conformed to the dentin surface and cured for a period of 40 seconds.

All samples were maintained for a week at room temperature in the physiologic serum and then cut buccolingually parallel to the longitudinal axis of the tooth via Universal Cutting Machine (Zwick, Ulam, Germany) so that at least two samples were obtained from each tooth (12 samples in each group).

Then, the interface between the dentin and restoration was grinded buccolingually and mesiodistally with the diamond grinding fissure bur number 012 (Tizkavan, Tehran, Iran) till the hourglass shape was created with 9μm dimensions in the buccolingual and mesiodistal sides to create a surface segment at the micron level in the area of tensile force implementation. Then, the samples were
adhered with cyanoacrylate adhesive (Zapit, Dental, USA) to the arms of Universal Testing Machine (model 160185, BISCO, Schaumburg, USA). The samples were subjected to 0.55 mm in a minute tensile force until they broke.

The microtensile strength was calculated using the following formula: With the calculation of force value (according to Newton’s law) and its division of the segment surface (based on the mm²), the results were reported in MPa. If the prepared samples were broken before the test conduction, number zero was recorded in the flowchart.

For a blind study, the person who conducted the tensile bond test only recorded the considered group number and was unaware of the types of application material in different groups. The assumption of normality was approved with the use of the Kolmogorov-Semirnov test (all \( p > 0.05 \)) while the homogeneity of variance was not approved via Levene’s test (\( p < 0.01 \)), so One-way ANOVA and Tamhane’s T2 tests were used for comparison of the groups; \( A p < 0.05 \) was considered as statistically significant.

**Results**

The One-way ANOVA test showed that there was a significant difference between the composite resin microtensile bond strength to the dentin among the groups (\( p < 0.001 \)). The average composite resin microtensile bond strength to the dentin is presented in Table1.

As shown in Table 1, the highest micro-tensile bond strength (31.86 ± 6.25 MPa) pertaining to group 2 where composite Filtek P90 was used with etch and bond on the dentin surface and the lowest was related to group 3 in which the composite Filtek Z250 was used with only bond and without etch on the dentin surface (11.95 ± 2.08). Tamhane’s T2 test showed that there was a significant difference between the micro-tensile bond strength of all other groups (\( p < 0.05 \)), except for between the first and fourth groups (\( p = 0.994 \)) (Table 1).

**Discussion**

For the microtensile bond strength conduction, the two methods of the sample construction beam and hourglass are used [18]. In the present study, the samples were prepared in the form of an hourglass and the reason for selection of this form was that beam preparation with accessible equipment and tools was not possible. Besides, in the case of the existence of the required equipment, also it seemed that beam preparation with a sufficient length in the primary teeth despite the pulp extent was difficult since in most of the samples the distance of the pulp to interphase was 3mm or less. On the other hand, hourglass form provides more attachment surface in the upper and lower areas of the designed arms of the test machine which reduces premature debond of the samples from the test machine arms [19-22].

In the present study, the microtensile bond strength test was used to assess the bond strength of the composites Filtek P90 and Filtek Z250 to the dentin of the primary teeth. For the comparison of the silorane composite, Filtek P90, with the methacrylate composites, Filtek Z250 had been used in a usual manner, which possesses a long record in relation to the newer methacrylate composites. Filtek Z250 is a micro-hybrid composite resin with 60% filler volume that has a polymerization contraction equivalent to 2.7 [23].

In order to investigate the surface etch effect on the micro tensile bond strength in one of the investigated groups, the dentin surface was etched with 37% acid phosphoric before applying silorane bonding system. For equivalence of the bonding factors, two kinds of

### Table 1: Mean value, standard deviation and \( p \)-Value of micro-tensile bond strength (MPa) of resin composite to dentin in different groups

| Groups                              | Mean ± SD    | \( p \)-value* |
|-------------------------------------|--------------|----------------|
|                                     |              | 1              | 2              | 3              |
| 1=(Siloran bond + composite Filtek P90) | 17.23 ± 3.47 | -              | -              | -              |
| 2=(Etch + siloran bond + Composite Filtek P90) | 31.86 ± 6.25 | 0.001          | -              | -              |
| 3=( single bond + Composite Filtek Z250) | 11.95 ± 2.08 | 0.001          | 0.001          | -              |
| 4=(Etch + single bond + Composite Filtek Z250) | 16.65 ± 2.50 | 0.994          | 0.001          | 0.002          |

*: Pairwise comparisons using Tamhane’s T2 post-hoc test
composites, for the Filtek Z250 composite the Single Bond, self etch bonding was used and in one of the investigated groups also the same bonding with the use of acid etch was adopted [22].

The present study results also showed that there was a significant statistical difference between the first and second groups in a manner that in the G2, in which acid etch was used before silorane bonding system, the microtensile bond strength was higher in comparison to the G1 in which only the silorane bonding system was used [23].

This fact can be explained in regard to the silorane bonding system to the teeth which has a two-bottle self-etch. The first stage was the use of a hydrophilic primer. Contrary to the primer systems of the two-step self-etch which are polymerized after bonding enforcement, in this primer the polymerization occurs in the first stage itself and prior to the bonding application; therefore, the bonding to enamel and dentin and formation of the hybrid layer take place in the first stage of the primer application similar to the one-step self-etch systems [24-27].

pH of the silorane primer is 2.7 till stability of the existing monomers in the primers and their half-life increases. This high pH of the primer classified silorane primer in the very weak (ultra-mild) group and silorane bonding contain two polar molecules attach from its hydrophilic front with hydrophilic primer and from its hydrophobia front to the silorane hydrophobia composite [28].

In the studies conducted by Mine et al. [29] on silorane bonding system, it was shown that the hybrid layer had a maximum of few hundred nanometers’ thickness that could be due to the high pH of the primer. In the grinded dentin with a very low agile, the hybrid layer thickness is in the extent of a few hundred nanometers.

Similarly, the resin tags are not seen due to the high pH of the primer; the smear plug is not removed from the inside of the dentin tubules. Therefore, in case the primer is implemented on a thicker layer from the smear layer which is produced via a quick diamond abrasive, the bond is created only in the superficial layer. So, the efficiency of the silorane bond is essentially in the first stage of the implementation of the primer and depends on the characteristics of the left smear layer.

Similarly, the composite Filtek Z250 with etch usage prior to the self etch bonding system showed better results because in the self etch systems with the use of the calcium and phosphoric ions primer that are separated from the apatite, the hydroxyl crystals will remain suspended in the alcohol and or the water that exists as a solvent in the primer. When this solution is evaporated with the use of the air aspiration, this calcium and phosphate content could reduce the execution solvency of the primer and lead to their sedimentation in the primer. This causes a decrease in the permeation ability of the bonding material to the surface prepared with a primer and eventually the bonding strength decreases [28].

The use of ‘acid etch’ prior to the primer implementation with elimination of a part of the calcium and phosphate ions prevents this from happening. Even though it is mentioned in some studies, that the strength of the bond systems, self etch and total etch does not have a statistically significant difference with each other, but the present study findings are not in the same line with this point [29].

Even the significant difference for the second and third groups can be related to the difference of the bond characteristics and the weaker bond of the self etch composite Filtek Z250 bond system in comparison to the silorane bond system after the acid etch usage. This can also be used to justify the structural difference of the two composites and a lower polymerization contraction of the silorane composite Filtek P90 in comparison to the methacrylate composite Filtek Z250 [30-31].

The recorded silorane polymerization shrinkage rate in the experimental studies [32-34] is lower than the entire methacrylate composites. Therefore, a lower force is imposed due to the contraction of the material during the strengthening on the interface of the material and dentin and this can be the reason for a closer contact of silorane with the surface and a better bond establishment [32-34].

Moreover, the other case that can have an effect on the bond strength to a dentin is the characteristics of dentin itself that is influenced by the age, tooth kind, decay rate, restorative or reaction dentin production and intermediate environment. For elimination of these variables in this study, the intact primary canines lacking decay in which less than 1/3 of their root length was eroded and had an equal intermediate level (serum) in the environment were used [35].

**Conclusions**

Considering the conducted study, the silorane resin composite (Filtek P90) had a higher micro-tensile bond strength to the primary dentin compared with
the methacrylate composite Filtek Z250, and etching the dentin surface prior to applying the primer and silorane bonding system leads to a better outcome.

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References

1. McDonald R, Avery D, Stookey G, et al. Dental caries in the child and adolescent. In: McDonald R, Avery D, Dean J. Dentistry for child and adolescent. 9th Edition. ST.Louis: Mosby M: 2011;177-204.
2. Moore K. Dental materials. In: McDonald R, Avery D, Dean J. Dentistry for child and adolescent. 9th Edition. ST. Louis: Mosby M: 2011;296-312.
3. Donly K, Segura A. Dental materials. In: Pinkham J, Casamassimo P, McTigue D, Fields H, Nowak A. Pediatric Dentistry Infancy through adolescence. 5th Edition. ST. Louis: Elsevier Saunders WB: 2013;325-340.
4. Dewaele M, Truffier-Boutry D, Devaux J, et al. Volume contraction in photocured dental resins: the shrinkage-conversion relationship revisited. Dent Mater. 2006;22: 359-365.
5. Charton C, Colon P, Pla F. Shrinkage stress in light-cured composite resins: influence of material and photo-activation mode. Dent Mater. 2007;23:911-920.
6. Cadenaro M, Biasotto M, Scuor N, et al. Assessment of polymerization contraction stress of three composite resins. Dent Mater. 2008;24:681-685.
7. Ilie N, Kunzelmann KH, Hickel R. Evaluation of micro-tensile bond strengths of composite materials in comparison to their polymerization shrinkage. Dent Mater. 2006;22:593-601.
8. Palin W, Fleming G, Burke F, et al. The influence of short and medium-term water immersion on the hydrolytic stability of novel low-shrink dental composites. Dent Mater. 2005;21:852-863.
9. Ilie N, Hickel R. Macro-, micro- and nanomechanical investigations on silorane and methacrylate-based composites. Dent Mater. 2009;25:810-819.
10. Van Ende A, Munck J, Mine A, et al. Does a low-shrinkage composite induce less stress at the adhesive interface? Dent Mater. 2009;25:825-833.
11. Nozaka K, Suruga Y, Amari E. Micro-leakage of composite resins in cavities upper primary molars. Int J Paediatr Dent. 1999;9:185-194.
12. Al-Boni R, Raja OM. Microleakage evaluation of silorane based composite versus methacrylate based composite. J Conserv Dent. 2010;13:152-155.
13. John R, Christense W, Henry W, et al. Space maintenance in the primary dentition. In: Pinkham J, Casamassimo P, McTigue D, Fields H, Nowak A. Pediatric Dentistry Infancy Through Adolescence. 5th Edition. ST. Louis: Elsevier Saunders: 2013. p. 379-384.
14. Stangel I, Nathanson D, Hsu CS. Shear strength of the composite bond to the etched porcelain. J Dent Res. 1987;66:1460-1465.
15. Sano H, Shono T, Sonoda H, et al. Relationship between surface area for adhesion and tensile bond strength evaluation of micro tensile bond strength of micro tensile bond test. Dent Mater. 1994;10:236-240.
16. Santos M, Podorieszach A, Rizkalla AS, et al. Microleakage and microtensile bond strength of silorane-based and dimethacrylate-based restorative systems. Compend Contin Educ Dent. 2013;34:19-24.
17. Kranjangta N, Sirsawasdi S. Micro tensile bond strength of silorane-based resin composite and corresponding adhesive in class I occlusal restoration. Am J Dent. 2011;24:346-353.
18. Cabrera E, Macorra JC. Micro tensile bond strength distributions of three composite material with different polymerization shirinkages to dentin. Adhes Dent. 2010;13:39-48.
19. Giacobbi MF, Vandewalle KS. Microtensile bond strength of a new silorane-based composite resin adhesive. Gen Dent. 2012;60:148-152.
20. Poureslami HR, Sajadi F, Sharifi M, et al. Marginal micro leakage of low shrinkage composite silorane in primary teeth. J Dent Res Dent Clin Dent Prospecot. 2012;6:94-97.
21. Soldo M, Simeon M, Matijevic J, et al. Marginal leakage of class V cavities restored with silorane-based and methacrylate-based resin systems. Den Mater. 2013;32:853-885.
tensile bond strength of different adhesive system to dent. KutipFak Derg. 2012;14:191-199.
23. Badr S, Atef-Ibrahim M, El-Seoud H. Silorane-based composite bond to primary and permanent teeth. Dent Mater. 2007;23:911-920.
24. Almeida e Silva JS, Rolla JN, Baratieri LN, et al. The influence of different placement techniques on the microtensile bond strength of low-shrink silorane composite bonded to Class I cavities. Gen Dent. 2011;59:233-237.
25. Pereira JDS, Dias CTS. Bond strength between silorane-based composite resin and dentin substrate. Am J Dent. 2013;4:90-94.
26. Fruits TJ, Duncanson MG Jr, Miller RC. Bond strength of fluoride releasing restorative materials. Am J Dent. 1996;9:219-222.
27. Daneshkazemi AR, Davari AR, Ataei E, et al. Effect of mechanical and thermal load cycling on micro tensile bond strength of clearfil SE bond to superficial dentin. Dent Res J. 2013;10:202-209.
28. Lien W, Vandewalle KS. Physical properties of a new silorane-based restorative system. Dent Mater. 2009;10:18-20.
29. Mine A, De Munck J, Van Ende A, et al. TEM characterization of a silorane composite bonded to enamel/dentin. Dent Mater. 2010;26:524-532.
30. Neelima L, Sathish ES, Kandaswamy D, et al. Evaluation of microtensile bond strength of total-etch, self-etch and glass ionomer adhesive to human dentin: An invitro study. Indian J Dent Res. 2008;19:129-133.
31. Koliniotou-Koumpia E, Kouros P, Dionysopoulo D, et al. Bonding strength of silorane based composite to Er-YAG laser prepared dentin. Lasers Med Sci. 2013;13:240-252.
32. Lowe RA. The search for a low-shrinkage direct composite. Inside Dent. 2010;1: 78-84.
33. Thalacker C, Heumann A, Weinmann W, et al. Marginal integrity of class V silorane and methacrylate composite restorations. J Dent Res. 2004;83:1364.
34. Bagis YH, Baltacioglu IH, Kahyaogullari S. Comparing microleakage and the layering methods of silorane-based resin composite in wide Class II MOD cavities. Oper Dent. 2009;34:578-585.
35. Ramos JC, Perdigao J. Bond strength and SEM morphology of dentin amalgam adhesives. Am J Dent. 1997;10:152-158.