Effect of Composite Elasticity and Filler Concentration on Shear Bond Strength of Composite to Dentin

Heshmat H. [a], **Alavi G.** [b], **Niknam M.** [b], **Alavi AA.** [c*]

[a] Dept. of Restorative Dentistry, School of Dentistry, Azad University, Tehran, Iran
[b] Dentist, Private Practice, Iran
[c] Dept. of Restorative Dentistry, School of Dental Medicine, Shiraz University of Medical Sciences, Shiraz, Iran

KEY WORDS

Composite;
Elasticity;
Shear strength;
Bovine;
Incisors;
Percentage.

ABSTRACT

Statement of problem: Resin composites are one of the most popular tooth colored restorative materials. Their enamel and dentin bonding ability are based on many factors, including elasticity and filler concentration.

Purpose: This study was undertaken to evaluate the effect of volumetric filler percentage and modulus of elasticity of six composites by measuring shear bond strength to dentin of the bovine teeth as experimental bonding substrate.

Materials and Methods: Eighty bovine incisors were prepared and divided into eight groups. Tetric Ceram, Tetric Flow, Compo glass F, Helimolar RO, Definite, and Degufill Mineral were applied respectively. A fifth generation of dentin bonding system [Single Bond (3M, USA)] was used for all the groups. The volumetric filler percentage of each composite was obtained from their specification data. The modulus of elasticity was determined, using the formula presented by Bream *et al.* (1986). Then, in last two groups Heliomolar RO and Definite were bonded using their own bonding systems, Syntac Multicomponent, and Definite Multibond. The shear bond strength was measured according to the ISO/TR 11405. Pearson's correlation test was used to compare the result with groups 4 and 5.

Results: The results indicated that the relationship between both the filler percentage and the modulus of elasticity with the shear strength was 16% ($P<0.005$). There was a significant difference between groups 4 and 7, but no significant difference was observed between groups 5 and 8.

Conclusion: Using composites with a higher filler content and modulus of elasticity resulted in higher shear bond strength in dentin bonding system. Our results showed that the corresponding dentin bonding system of composites had better results.

Received Oct. 2008;
Received in revised form Jan. 2009;
Accepted Nov. 2008

* **Corresponding author.** Alavi AA., Address: Dept. of Restorative Dentistry, School of Dental Medicine, Shiraz University of Medical Sciences, Shiraz, Iran; Tel: 711-6307245; Fax: 711-5270325; E-mail: alavia@sums.ac.ir

Introduction

When Bunocore introduced Acid-etch technique in 1955, a revolution occurred in esthetic dentistry. Thereafter, enamel and dentin adhesions were studied and many manufacturers tried to produce different bonding systems. In addition to enamel and dentin bonding, bonding to amalgam, porcelain and metal are also available. Now we are in the adhesive dentistry era, when conservative restorations have replaced the extensive and mechanically retentive cavity preparations. Increasing demand for esthetic restorations has led to production of many types of composites. High elasticity and increased filler concentration would increase bond strength to the enamel [1]. The effects of these factors on bond to dentin are not yet clear.

Comparison of the in vitro studies to evaluate the DBAs (Dentin bonding Agents) is very difficult and causes many ambiguities in evaluating their clinical results [2]. It may be due to the differences in technical sensitivities of these systems as well as varieties in the composite materials [3]. In some studies, the bovine teeth were used as an experimental model [4,5,6]. They have been provided in a short period of time. So, they could be controlled to be caries-free. The infection control in the bovine teeth is not as important as in human's teeth. So, the effect of disinfectant agents on the bond is minimal. The shear bond strength in human and the bovine teeth has no significant statistical difference and the mean bond strength in the bovine teeth is lower than that of human [4,5].

Researchers have carried out a variety of investigations to determine the properties of available dentin bonding systems [7]. Measurement of dentin bond strength is a sensitive procedure in clinical and laboratory studies. Some researchers prefer using the same brand of bonding agent and composite.

Composite resins could be classified based on filler size, filler concentration and their composition [8]. Hasegawa *et al*. reported that high bond strength could be obtained with better mechanical properties of composite resin [9]. Miyazaki *et al*. has also indicated that the initial setting behavior of bonding agents containing filler particles may be one of the important factors influencing the dentin bond strength [10]. On the other hand, Schneider *et al* in their study showed that the difference in the composite filler type and amount had little influence on the bond strength [11].

In this study, based on Braem mathematic formula ($E=3103.33e^{0.029771720X}$, E=Youngs modulus, X=Volumetric filler concentration) the elasticity of the composite due to volumetric filler concentration was calculated, and also the shear bond strength of six types of composites was evaluated according to the standard report of ISO/TR 11405. The statistical correlation between the elasticity and volumetric filler concentration on shear bond strength was determined using Pearson's correlation test.

Materials and Methods

Eighty bovine incisor teeth were sectioned horizontally in order to prepare flat dentinal surfaces and then randomly divided into 8 groups of 10 teeth. The first six groups were evaluated for filler percentage and elasticity affecting the shear bond strength by using six different composites and one bonding agent. The last 2 groups were two composites with their own bonding systems. To confine the bonding area, a Teflon Split mold (3 mm internal diameter and 4 mm height) was prepared and to stabilize the Teflon mold on the dentin surface, putty and a clip were applied and the internal walls were soaked in Vaseline to prevent the composite from sticking to mold walls. 37% orthophosphoric acid was applied on the dentinal surfaces for 15 seconds, and rinsed with water for 10 seconds. The excess water was removed and air dried for 5 seconds and a surface with a relative humidity was obtained. Single Bonds (3M, USA) were used as bonding agents on the dentin surface and cure for 10 seconds and the bonding processes were repeated in groups 1 to 6 (Table 1).

Table 1 Groups studied and materials used

Group	Bonding agent	Composite
1	Single Bond	Tetric Ceram
2	Single Bond	Tetric Flow
3	Single Bond	Compo glass
4	Single Bond	Heliomolar RO
5	Single Bond	Definite
6	Single Bond	Degufill Mineral
7	Syntac Multicomponent	Heliomolar RO
8	Definite Multibond	Definite

The composites were put in mold cavities and light cured for 60 seconds. After splinting, the Teflon mold composite was light cured for an additional 40 seconds. In groups 7 and 8, the Syntac Multicomponent (Vivadent, Lichtenstein) and Definite Multibond (Degussa, Germany) were used as bonding agents for Heliomolar and Definite, respectively (Tables 2, 3).

The specimens were restored in tap water for 24 hours in room temperature. Thermo-cycling was done between 05-50c for 500 cycles. To determine the shear bond strength, the Nogoshi shear apparatus which was made up of a metal body inside was used and a cylindered hole (26 mm diameter and 12 mm thickness) was designed to fix the samples. Pressure was applied using a sliding plate, which had three holes of 3, 5 and 10 mm diameter depending on the type and the diameter of the material used for the cylinder base. The blade's thickness was designed to be 1 mm and placed between the plate and the body. The pressure was just parallel with the boundaries between the composite and the dentin in the bond area. The applied pressure in the shear was fixed in a tension-compression apparatus and the dentin samples were put in their specific sites which finally caused a shear stress. The composite filler percentage was determined by its guiding instructions and the composite elasticity coefficient was calculated using $E=3103.33e^{0.029771720X}$ formula.

To compare the bond strength in the first six groups, ANOVA was used for statistical analysis. To find any correlation between the filler concentration and the composite elasticity coefficient with shear bond strength, the Pearson's Correlation and Multiple Regression tests were used. $(a = 0.05)$ The samples were studied under a light microscope (40X) and failures were listed as adhesive failure in the border between the dentin and bonding agent, incomplete adhesive failure in the border between the composite and bonding agent, cohesive failure and crack in dentin, and cohesive failure in the composite.

Table 2 Composite types and their characteristics

Composite	Groups (on filler type aspect)	Filler weight percentage	Volumetric filler percentage	Filler type	Filler size (mm)	Composition
Tetric Ceram	Fine-Particle Hybrid	80	60	Barium glass Ytterbium trifluorideBa-Al-fluorosilicateglass, Highlydispersed silicon dioxide Spheroid mixed oxide	0.04-3	Bis-GMA Urethane dimethacrylate Triethylene glycole dimethacrylate
Heliomolar RO	Micro fill	66.7	46	Silicondioxide Ytterbium trifluoride Copolymer	0.04-02	Bis-GMA Urethane dimethacrylate
Tetric Flow	Fine-Particle Hybrid	68.1	43.8	Bariumglass Ytterbium trifluroideBa-Al-fluorosilicateglass Highlydispersed silicon dioxideSpheroid mixed oxide	0.04-3	Bis-GMA Urethane dimethacrylate Triethylene glycole dimethacrylate
Compo glass F	N/A	77	55	Ytterbium Trifluoride Ba-Al-Fluorosticate glass Spheroid mixed oxide	0.2-3	Urethane dimethacrylate Teraethylene glycole dimethacrylate Cycloatiphatic Dicarboxylic acid dimethacrylate
Degufill mineral	Ultra-Fine Hybrid	N/A	62	Ba-Al-Borosilicate Ca phosphate- Fluorid-Apatite	N/A	Bis-GMA Methacrylates Initiator Stabilizer
Definite	N/A	77	60	N/A	N/A	Polymerizable Ormocer-matrix Inorganic fillers Initiators, stabilizer, pigments

Table 3 Bonding systems with their compositions and their processes

Bonding type	Bonding process (after cavity preparation)	Composition
Single bond (3M)	1- Etching for 15 seconds. 2- Rinsing with water for 10 seconds and getting extra water 3- An adhesive layer was applied on enamel and dentin and slow flow of air for 2-5 seconds. 4- Light for 10 seconds. 5- Repeating steps 3&4	N/A
Definite Multibond (Degussa)	1- Etching for 15 seconds. 2- Rinsing with water for 15-30 seconds and getting extra water with air 3- Applying primer for 30 seconds and slow flow of air. 4- Applying adhesive for 10 seconds and then light for 10 seconds.	**Primer** Ormocer matrix Ethanol, H2O, mono and dimethacrylates Initiators and stabilizers **Adhesive** Ormocer matrix Mono and dimethacrylates and polymers Glass fillers Initiators and stabilizers
Syntac Multicomponent (Vivadent)	1- Etching for 10-15 seconds, rinsing with water and complete drying 2- Applying primer and drying with air after 15 seconds. 3- Applying adhesive and drying with air after 10 seconds. 4- Applying bonding agent, aerating and light for 10 seconds.	**Primer** Tetra ethylene glycoldimethacrylate 0.25g Maleic acid in watery acetone solution 0.04g **Adhesive** Polyethylene glycoldimethacrylate 0.35g Glutaraldehyde 50% in watery solution 0.10g

Results

A significant difference was observed between all the groups in relation to shear bond strength (Table 4) and there was a significant correlation between the shear bond strength and the filler concentration and composite elasticity coefficient (r^2=0.16, P<0.005) (Table 5). The effect of Filler concentration and elasticity on the shear bond strength was 16% (Fig 1, 2). The differences between groups 4 and 7 and between groups 5 and 8 were significant (Tables 4). The results of the microscopic investigation (40 X) are shown in Table 6. E type failure was not found in any of the samples but A type failure was the most one observed (35%).

Table 4 Shear bond strength with elasticity coefficient and filler percentage of the 8 groups

Group (n=10)	Meran shear bond strength (Mpa)	Standard deviation	Volumetric filler percentage	Elasticity coefficient Gpa (calculated by bream formula)
1	18.75	5.46	60	18.5
2	17	3.33	43.8	11.5
3	15.39	4.99	55	16
4	10.85	3.44	46	12.2
5	18	4.75	60	18.5
6	19.49	5.54	62	20
7	18.9	5.6	46	12.2
8	19.8	2.71	60	18.5

Table 5 Correlation between shear strength and volumetric percentage and elasticity coefficient in the first 6 groups

	1	2	3	4	5	6	
Mean shear strength	18.75	17	15.39	10.85	18	19.49	r^2=0.16 P<.005
Elasticity coefficient	18.5	11.5	16	12.2	18.5	20	
Volumetric percentage	60	43.8	55	46	60	62	

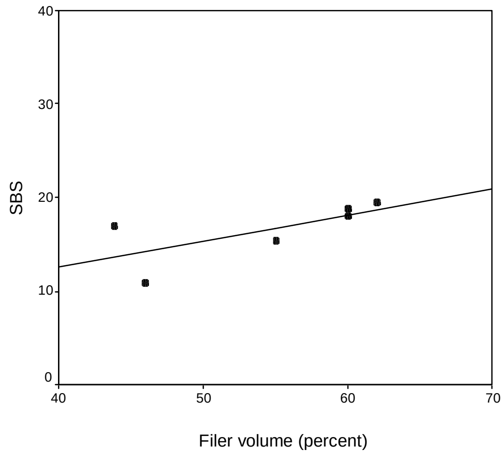

Figure 1 Correlation between volumetric filler percentage and shear bond strength of the first 6 groups of composites

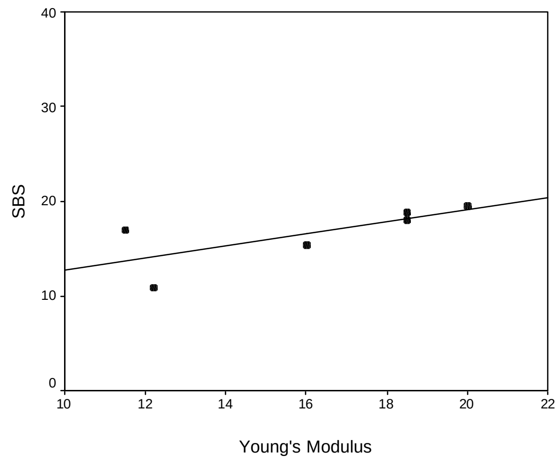

Figure 2 Correlation between elasticity coefficient and shear bond strength of the first 6 groups of composites.

Table 6 Microscopic results with specifying group and failure types. (A: Adhesive failure in the boundary of dentin and bonding agent, B: Adhesive failure, C: Adhesive failure in the boundary of composite and bonding agent, D: Cohesive failure in the dentin and E: Cohesive failure in composite)

Group n=10	A	B	C	D	E
1	5	1	-	4	-
2	7	3	-	-	-
3	1	6	3	-	-
4	-	4	6	-	-
5	4	3	1	2	-
6	-	7	-	3	-
7	6	1	-	3	-
8	5	-	-	5	-
Total	28	25	10	17	0

Discussion

In the first 6 groups, single bond dentin adhesive was used from the 5th generation. Then six types of composites were used which had different volumetric percentages of filler concentrations. As Nano fillers and their bonding condition are still under investigation, we preferred to use a bonding system without filler. In groups one and three, Tetric Ceram and Tetric Flow with similar ingredients, except for filler percentages were used (Table 2). The results showed that the shear bond strength was independent of the filler concentration.

Also, in other groups the same results were obtained. The interesting points were that both Tetric Ceram and Definite had 60% filler concentration and their shear bond strength was almost the same. These results confirmed the effect of filler percentage on the shear bond strength value. In microfine composites, the bond strength to the dentin was approximately 10-17 Mpa while the bond strength to the enamel was 30 Mpa. In fine composites, the bond strength to the dentin was 5-24 Mpa while it was 34 Mpa for the enamel. This wide range of bond to the dentin could be attributed to the differences in the biological effects, clinical situations, laboratory studies and methods of measuring shear bond strength [12,13,1]. Boyer *et al*. (1982) in a study surveyed the filler concentration effect of microfilled and hybrid composites on the shear bond strength to the enamel. The result showed higher fillers, resulting in more tensile bond strength with no effect on elasticity and a decrease in the polymerization shrinkage and linear expansion coefficient of the composite [1].

Among mechanical properties of the composites, Young's modulus was dependent on volumetric filler percentage. Our composite filler percentage was located in Bream's domain and the filler sizes were nearly similar and comparable. So, the Bream formula could be attributed to our samples. Since this correlation was exponential, there was a possibility that the filler percentage and elasticity coefficient had no similar statistical correlation with the shear bond strength. Pearson's correlation was used for evaluation of this relation. As r^2=0.16%, the filler and elasticity effects on the shear bond strength was 0.16%. Boyer *et al* and Van-Noort in their studies reported that bonding strength of different materials to the dentin had several determinants such as the elasticity constant, which had more effects on the shear bond strength. The more the elasticity coefficient was, the stiffer was the composite and if the composites were used with a higher elasticity coefficient, the bond strength might be doubled [1,15].

If the composites used in the stress area had a lower elasticity, they would show an earlier deformation very soon which would increase the possibility of micro-leakage and it would be better not to use the microfilled composites in the posterior teeth [8]. As in the wedge-shaped cavities, the gingival floor would provide a wider angle toward occlusal force and the shear forces would increase the longevity of the restoration [14]. Fine particle composites with higher elasticity would stand against the orthodontic forces better than the micro-fine ones [12].

Our results showed that although the tetric flow group had the least elasticity coefficient, the filler percentage showed a 17±3 Mpa bond strength. This could be due to the adaplibity of the composite resin with bonding agent, because of its high flow and elasticity. Pashley *et al* stated that bending of the composites may occur during shear bond strength tests [16]. So, the elasticity of Tetric flow during application of shear forces could be partially due to this bonding effect. The particle size in this resin, like Tetric Ceram which was a fine particle hybrid, and the decreasing filler percentage had a direct effect on the concentration and flow of the composite. Bond strength could change as a result of filler percentage of the filled adhesive [13]. The acceptable bond strength is 17 Mpa for a cavity with C.factor equal or less than one [15]. The four types of composite, i.e. Tetric Flow, Tetric Ceram, Mineral and Definite, produced acceptable bond with the Single Bond and from the clinical aspect, application of these composites in combination with a 5th generation of bonding system was relatively acceptable. To compare this bonding agent with bonding system recommended by the manufacturer and the bonding, two groups were considered in our study as Heliomolar RO group which showed a significant bond difference with the Single Bond and with the suggested bonding system which was Syntac multicomponent, and the Definite group, with no significant difference between the single bond and Degufill Multibond. The reports of Leirskar *et al*. showed that the differences in the results using composites with different bonding systems were due to physical and mechanical properties of the composite, including the physical absorption, interfacial diffusion and mecha-nical interlocking [3]. So, it would be better to use the composite and its own bonding of one brand [2]. In Heliomolar composites, in addition to filler concentration and their sizes, the compatibility with bonding system was very important. So, using the composites with different bonding systems was not always possible.

In 40% of the samples in group 1, 20% of the samples in groups 5, 30% of those in groups 6 and 7 and 50% of those in group 8, some cracks were evident in the dentin that might be satisfactory for the researcher as an evidence of strong bond. Two theories may describe the dentin weakening as existence of hybrid layer in the dentin, and weakening of the dentin due to dehydration [16].

Also, when the resin bond was confined in the area under the composite cylinder, the stress in the dentin could be more than that produced in the bond margin, so, dentin failure would be probable. In other words, always cohesive fracture in the dentin does indicate that bond strength is more than the dentin strength. Usually, the composite cylinder with higher elasticity would produce dentinal cracks in a lower force [16]. Most of the restorative materials are more resistant to compressive rather than tensile forces. In shear strength tests, the composites are more under compressive stress (except in cylinder in which the stress is in tensile form); meanwhile, the stress in the dentin is manifested in tensile form [17]. Since the tensile strength in the dentin was lower than the composite compressive force, the crack pattern tended toward the dentin. In Heliomolar and Compo-glass F group, most of the failures occurred in the composite and bonding agent margins showing the incompatibility of the composites with the bonding agent. In samples containing incomplete failure in the margin of the restorations, the failure pattern was semilunar in circumference of the bond area, so the stress concentration was on one side of the composite cylinder which was similar to the results of the study by Versluis [17].

Conclusion

Using composites with a higher filler content and modulus of elasticity resulted in higher shear bond strength in the dentin bonding system. Our results in groups 4, 7 indicated that the corresponding dentin bonding system of the composites revealed better results.

Acknowledgements

The authors would like to thank the Office of Vice-Chancellor for Research of Shiraz University of Medical Sciences for financial support.

References

[1] Boyer DB, Chalkley Y, Chan KC. Correlation between strength of bonding to enamel and mechanical properties of dental composites. J Biomed Mater Res 1982; 16: 775-783.

[2] Oilo G. Bond strength testing--what does it mean? Int Dent J 1993; 43: 492-498.

[3] Leirskar J, Oilo G, Nordbø H. In vitro shear bond strength of two resin composites to dentin with five different dentin adhesives. Quintessence Int 1998; 29: 787-792.

[4] Retief DH, Mandras RS, Russell CM, Denys FR. Extracted human versus bovine teeth in laboratory studies. Am J Dent 1990; 3: 253-258.

[5] Schilke R, Bauss O, Lisson JA, Schuckar M, Geurtsen W. Bovine dentin as a substitute for human dentin in shear bond strength measurements. Am J Dent 1999; 12: 92-96.

[6] Ishii T, Ohara N, Oshima A, Koizumi H, Nakazawa M, Masuno T, Matsumura H. Bond strength to bovine dentin of a composite core build-up material combined with four different bonding agents. J Oral Sci 2008; 50: 329-333.

[7] al-Salehi SK, Burke FJ. Methods used in dentin bonding tests: an analysis of 50 investigations on bond strength. Quintessence Int 1997; 28: 717-723.

[8] Lambrechts P, Braem M, Vanherle G. Buonocore memorial lecture. Evaluation of clinical performance for posterior composite resins and dentin adhesives. Oper Dent 1987; 12: 53-78.

[9] Hasegawa T, Itoh K, Koike T, Yukitani W, Hisamitsu H, Wakumoto S, *et al*. Effect of mechanical properties of resin composites on the efficacy of the dentin bonding system. Oper Dent 1999; 24: 323-330.

[10] Miyazaki M, Ando S, Hinoura K, Onose H, Moore BK. Influence of filler addition to bonding agents on shear bond strength to bovine dentin. Dent Mater 1995; 11: 234-238.

[11] Schneider BT, Baumann MA, Watanabe LG, Marshall GW Jr. Dentin shear bond strength of compomers and composites. Dent Mater 2000; 16: 15-19.

[12] Craig RG, Powers JM. Restorative Dental Materials. 11th ed. St. Louis: C.V. Mosby; 2002. p.246-248.

[13] Van Meerbeek B, Perdigão J, Lambrechts P, Vanherle G. The clinical performance of adhesives. J Dent 1998; 26: 1-20.

[14] Van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. J Dent 1989; 17: 61-67.

[15] Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. Dent Mater 1995; 11: 117-125.

[16] Unterbrink GL, Liebenberg WH. Flowable resin composites as "filled adhesives": literature review and clinical recommendations. Quintessence Int 1999; 30: 249-257.

[17] Versluis A, Tantbirojn D, Douglas WH. Why do shear bond tests pull out dentin? J Dent Res 1997; 76: 1298-1307.