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Flexural Strength and Microshear Bond Strength of Conventional and Flowable Composite Resins to Dentin

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Abstract

Background and Aim: This study aimed to evaluate the flexural strength (FS) and microshear bond strength (μ SBS) of conventional and flowable composite resins to dentin.

Materials and Methods: This in vitro study was conducted on composite specimens fabricated from Filtek Z250 and Clearfil AP-X Esthetics (CE) conventional, and Clearfil AP-X Esthetics Flow (CEF) and Clearfil AP-X Flow (CF) flowable composite resins in 4 groups (n=10). The specimens underwent a 3-point bending test for measurement of their FS. For assessment of μ SBS, dentin discs with 2 mm thickness were obtained from the coronal third of the crowns of 20 extracted premolars. Composite resins were bonded to dentin discs in Tygon tubes (1 mm height, 0.7 mm internal diameter) using Tetric N-Bond 5th generation bonding agent, and a universal testing machine measured their μ SBS. Data were analyzed by ANOVA and Tukey test (alpha=0.05).

Results: Z250 showed a significantly higher FS than CEF (P=0.002); no other significant difference was found in FS. The two conventional composites (P=0.847) and the two flowable composites (P=0.575) had no significant difference with each other in μ SBS. However, the conventional composites had a significantly higher μ SBS than the flowable composites (P<0.001).

Conclusion: The FS of CEF was significantly lower than that of Z250, and comparable to that of other tested composite resins. All tested composites had a FS higher than 80 MPa recommended by ISO-4049 for restorations under occlusal forces. Both CEF and CF showed significantly lower μ SBS than the tested conventional composite resins. **Keywords:** Composite Resins; Dental Bonding; Flexural Strength

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Introduction

Composite resins were introduced in response to patient demands for tooth-colored restorations [1]. They require less tooth

preparation, do not have thermal and electrical conductivity, and are more esthetically pleasant than amalgam restorations [2].

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The bond strength of composite to tooth structure is an important parameter in success of composite restorations. For an ideal bond strength, close contact of the substrate and adhesive is imperative. Also, the surface tension of adhesive should be lower than the free surface energy of the substrate [3]. In the etch-and-rinse systems, adhesion is based on resin penetration between the unsupported collagen fibers after smear layer removal, and subsequent formation of a hybrid layer [4]. However, in self-etch systems, optimal bond strength is achieved by etching and simultaneous penetration of acidic monomers that play a key role in resin-dentin adhesion, and do not completely dissolve the smear layer and mineral dentin components.

Dentin bonding depends on stress and tension at the resin-dentin interface and properties of the hybrid layer [5]. Several clinical methods have been proposed to achieve a high bond strength, such as incremental application of composite and increasing the contact time of dental substrate and the bonding agent; these methods improve the quality of the hybrid layer [6]. The size of specimen, type of composite, and type of test can affect the bond strength [7]. Also, lower filler content to increase flowability of composite can degrade the physical and mechanical properties of composite resins, such as their microshear bond strength (µSBS) [8].

Flexural strength (FS) is another important property of restorative materials that depends on material composition, environment, and failure mode. Structural defects in composite materials can also decrease their FS, and lead to microleakage and gap formation [9-11]. Evidence shows that nano-size filler particles can increase the FS [12]. Also, flowable composites have shown fewer voids and lower leakage than other composite types in some studies [2, 13, 14] while some others found no significant difference [2, 15]. In general, flowable composite resins have a FS lower than that of conventional composite resins by approximately 20%, mainly due to their lower filler content [16]. Thus, they should not be used in extensive cavities under heavy occlusal forces [2]. Also, lower filler content of flowable composite resins can increase their polymerization shrinkage, which increases the stress level, lowers the FS, and can eventually lead to restoration fracture [17].

The manufacturer of Clearfil AP-X Esthetics Flow (CEF), as a newly introduced flowable composite, claims that it has optimal mechanical properties for restoration of Class II cavities due to high filler content and silanization of its fillers. However, information in this regard is lacking. Thus, this study aimed to assess the FS and μ SBS of Filtek Z250 and Clearfil AP-X Esthetics (CE) conventional, and CEF and Clearfil AP-X Flow (CF) flowable composite resins to dentin. The null hypothesis of this study was that no significant difference would be found in FS and μ SBS of the tested conventional and flowable composite resins to dentin.

Materials and Methods

This in vitro, experimental study was conducted on 4 types of composite resins including 2 flowable composite resins, namely CEF and CF, and two conventional composite resins namely Filtek Z250 and CE (Table 1). Also, 20 extracted premolars were used for preparation of dentin discs for measurement of µSBS. The study protocol was approved by the ethics committee of the university (IR.SBMU.RIDS.REC.1396.608).

Sample size:

The sample size was calculated to be 10 in each group considering the presence of 4 independent groups using one-way ANOVA feature of PASS 11 assuming alpha=0.05, beta=0.2, study power of 80%, and mean FS of 76 MPa, 87 MPa, 55 MPa, and 88 MPa in the four groups with a standard deviation of 14 according to a previous study [18].

Group	Composite	Manufacturor	Organic matrix	Inorganic filler	Filler size	Filler volume	Filler weight
	resin	Manufacturer			(µm)	percentage	percentage
A	Clearfil AP- X Esthetics Flow	Kuraray Noritake Dental Inc., Japan	TEGDMA	Silanated- barium glass Colloidal silica	0.18-3.5	48-62%	75%
В	Clearfil AP-X Flow	Kuraray Noritake Dental Inc, Japan	TEGDMA	Silanated- barium glass Silica	0.02-19	62%	81%
С	Filtek Z250	3M ESPE, St Paul, MN, USA	Bis-GMA UDMA Bis-EMA	Zirconia/Silica	0.01-3.5	60%	82%
D	Clearfil AP-X Esthetics	Kuraray Noritake Dental Inc, Japan	Bis-GMA	Silanated- barium glass PPOF*	Unclear	66%	78%

Table 1. Characteristics of the materials used in this study

*Pre-polymerized organic filler

Specimen fabrication for the 3-point bending test:

The 3-point bending test was performed according to ISO-4049. A two-piece stainlesssteel mold measuring 2 x 2 x 25 mm was used for this purpose. The two pieces were placed next to each other and fixed with an adhesive tape. The assembly was then placed on a clean glass slab, and composite resin was applied into the mold. A transparent polyester matrix band (TDV Dental Ltd., Germany) with 0.05 mm thickness was placed over it, and gently compressed with a glass slab in order for the excess composite to leak out. Each specimen was cured from 3 directions of left, right, and middle by a light curing unit (Demetron LC, SDS Kerr, Germany) for 20 seconds per each direction (a total of 60 seconds). The tip of the curing unit was directly placed on the glass slab. The center was light-cured first and then the left and right sides. The specimens were removed from the mold and incubated in distilled water at 37°C (PECO Incubator PI-455G, Iran) for 24 hours. The dimensions of each specimen were then measured by a digital caliper (Mitutoyo, Japan). The FS of the specimens was then measured in a universal testing machine (Z020; Zwick Roell, Ulm, Germany) with three parallel rods with a circular cross-section with 2 mm diameter. Two rods were in the lower compartment with 20 mm distance from each

other, and the third rod was in the upper compartment at the midpoint of the distance between the two lower rods. Load was applied at a crosshead speed of 0.5 mm/minute according to ISO-4049 and continued until specimen fracture. The load at fracture was recorded in Newtons (N), and FS in megapascals (MPs) was calculated using the following formula:

$\sigma = 3FI/2bh^2$

Where F is the maximum load in Newtons, b is the specimen width in millimeters, I is the distance between the two supports in millimeters, and h is the specimen height in millimeters.

Specimen preparation for µSBS test:

Twenty freshly extracted sound premolars were immersed in 0.2% thymol (Merck, Germany) solution for 48 hours for disinfection. They were then stored in saline at room temperature. The time interval between the extraction and the experiment was less than 2 months. The teeth were sectioned by a diamond disc (Bosch, Germany) to eliminate the occlusal enamel and expose the underlying dentin. The upper surface of the sections was etched with 37% phosphoric acid (Morva Etch, Morvabon, Iran), rinsed with water spray after 10 seconds, and dried with air spray. One layer of bonding agent (Tetric N-Bond; Ivoclar Vivadent, Schaan,

147 Farahiparizi et al.

Liechtenstein) was applied on each specimen, and air-sprayed in order for the solvent to evaporate. Light curing was then performed for 20 seconds using a light-curing unit (Demetron LC, SDS Kerr, Germany) with a light intensity of 600 mW/cm². Tygon tubes with an internal diameter of 0.7 mm and 1 mm height were filled with the respective composite, placed on dentin discs, and cured for 20 seconds. Five dentin discs were used for each composite group, and two Tygon tubes (Miami Lakes, FL, USA) were bonded to each disc (n=10 specimens for each composite type) (Figure 1). The specimens were then incubated at 37°C for 24 hours.



Figure 1. Composite cylinders bonded to dentin disc for measurement of $\ensuremath{\mu SBS}$

After 24 hours, the Tygon tubes were cut by a scalpel. The specimens were fixed to the jig of a microtensile tester (Quantrol by Dillon, USA) by cyanoacrylate glue (Jalafix 123, Jalasanj Company, Iran). A wire was tied around the cylinder, and load was applied until debonding. The load causing debonding was recorded in Newtons (N). The following formula was used for its conversion to megapascals (MPa):

µSBS (MPa)=N/mm²

The diameter of all specimens was 0.7 mm. Thus, the surface area of all specimens was calculated to be 0.384 mm².

Statistical analysis:

Normal distribution of data was ensured by the Kolmogorov-Smirnov test, while equality of the variances was confirmed by the Levene test. Thus, FS and μ SBS of the four groups were compared by one-way ANOVA, followed by pairwise comparisons with the Tukey test. All statistical analyses were performed using SPSS version 20 (SPSS Inc., IL, USA) at 0.05 level of significance.

Results

FS:

Table 2 presents the measures of central dispersion for the FS of the four groups. Filtek Z250 showed the highest, and CEF showed the lowest mean FS. The four groups had a significant difference in FS (P=0.004). Pairwise comparisons showed that the FS of Z250 was significantly higher than that of CEF (P=0.002). No other significant differences were found (P>0.05). μSBS :

Table 3 presents the measures of central dispersion for the μ SBS of the four groups. The four groups had a significant difference in μ SBS (P<0.001). Pairwise comparisons showed no significant difference in μ SBS of the two flowable (P=0.575), and the two conventional (P=0.847) composite resins. However, the two conventional composite resins showed a significantly higher μ SBS than the two flowable composite resins (P<0.001).

Table 2. Measures of central dispersion for the FS (MPa) of the four groups

Group	Mean	Std. deviation	95% CI		Minimum	Mavimum
Group			Lower bound	Upper bound	Millinum	Maximum
Clearfil AP-X Esthetics Flow	133.92a	27.57	114.20	153.65	83.25	174.12
Clearfil AP-X Flow	149.70ab	15.02	138.96	160.45	123.66	174.30
Filtek Z250	174.48b	20.79	159.60	189.36	133.47	208.25
Clearfil AP-X Esthetics	157.97ab	27.47	138.31	177.62	128.25	231.00

*Similar letters indicate absence of a significant difference (P>0.05).

Group	Mean	Std. deviation	95% CI		Minimum	Marian
Group			Lower bound	Upper bound	Minimum	Maximum
Clearfil AP-X Esthetics Flow	10.02b	2.34	8.34	11.70	6.51	14.58
Clearfil AP-X Flow	9.03b	1.53	7.93	10.13	7.81	13.02
Filtek Z250	16.95a	1.40	15.94	17.95	14.84	19.01
Clearfil AP-X Esthetics	17.57a	1.37	16.58	18.55	15.62	19.79

Table 3. Measures of central dispersion for the μSBS (MPa) of the four groups

*Similar letters indicate absence of a significant difference (P>0.05).

Discussion

This study assessed the FS and μ SBS of four conventional and flowable composite resins to dentin. The results showed that Z250 had a significantly higher FS than CEF. Also, the conventional composites had a significantly higher μ SBS than the flowable composites. Thus, the null hypothesis of the study was rejected.

The manufacturer of CEF claims that it has excellent mechanical and physical properties and may be reliably used for restoration of Class I and II cavities. All tested composites in this study except for Z250 have silanated fillers. The results regarding FS showed that the mean FS of all tested composites was higher than the minimum required threshold of 80 MPa according to ISO-4049 [19]. The FS of Z250 was significantly higher than that of CEF but no other significant differences were found. High FS protects the restoration against fracture and preserves the tooth structure as such [20]. Previous studies attributed the lower mechanical and physical properties of flowable composites compared with conventional composites to their lower filler content [17, 21, 22]. However, in the present study, the filler volume of both tested flowable composites was comparable to the filler volume of the tested conventional composites. Thus, this difference may be attributed to different types and composition of monomers used in Z250 and CEF. Z250 has a combination of bis-GMA, UDMA copolymer, and TEGDMA and bis-EMA comonomers while CEF only contains TEGDMA. Since CEF is a flowable composite with high filler volume, it requires a low molecular weight monomer in order not to increase its viscosity and also in order to allow a high filler content [23]. TEGDMA has a lower molecular weight than bis-GMa and is therefore used as a diluting agent to lower the viscosity of composite resins and allow for incorporation of a higher filler content [23-26]. However, it lowers the mechanical properties such as FS of composite resins due to its lower molecular weight and higher number of double bonds [26, 27]. It has been reported that composite resins with a combination of bis-GMA, UDMA, and TEGDMA monomers have superior physical and mechanical properties compared to those that only contain TEGDMA [28].

CEF, CF, and CE all have silanated fillers. However, no information is available regarding the silanization process of their fillers. In general, information about the effect of silanization on composite properties is controversial. Some studies showed that silanization of fillers increased their adhesion to the matrix and resulted in more homogenous dispersion of fillers in the matrix, leading to improvement of physical and mechanical properties such as FS compared with composites devoid of silanated fillers [29-31]. However, Beatty et al. [32] demonstrated that addition of silane to filler significant surface had no effect on physicomechanical properties. Also, it has been stated that silane can decrease homogenous dispersion of fillers due to formation of nonhomogenous granules [33]. Since the three tested composites with silanated fillers had no significant difference in FS, it may be stated that silanization of fillers in flowable composites can improve their FS to the level of a silanated conventional composite; however, information is not sufficient for comparison with a nonsilanated conventional composite (i.e., Z250), and further investigations are required in this regard.

Assessment of µSBS in the present study revealed no significant difference in µSBS of the two flowable and the two conventional composite resins. However, the two conventional composite resins showed a significantly higher µSBS than the two flowable composite resins. Both flowable composites tested in the present study are highly filled as stated by the manufacturer. Highly-filled flowable composites reportedly have a lower polymerization shrinkage and lower stress at the interface, and therefore, yield higher bond strength. However, studies found no significant difference in bond strength of highly-filled and normally-filled flowable composites [34, 35]. Also, the present study showed lower µSBS of flowable composites than conventional composites. The same results were reported by Sagsoz et al. [35] who showed that higher filler content of flowable composites did not cause a higher bond strength to dentin compared with the conventional and bulk-fill composite resins. On the other hand, it has been widely reported that stresses due to polymerization shrinkage and their concentration at the composite-tooth interface are responsible for debonding [8, 35, 36]. This shrinkage mainly depends on monomer composition, type and volume of fillers, and the initiation mechanism of polymerization reaction [36]. As mentioned earlier, flowable composites tested in the present study contain TEGDMA, which increases the degree of conversion and subsequently the polymerization shrinkage [26, 37]. Also, according to Alonso et al, [38] TEGDMA causes greater stress in composite resins due to the presence of aliphatic group in its structure [38, 39]. Thus, the presence of TEGDMA in flowable composites evaluated in the present

study may explain their lower µSBS than that of conventional composites.

In vitro design and no simulation of the clinical setting by aging were the main limitations of this study, which should be addressed in future studies.

Conclusion

According to the results of this study, the FS of CEF was significantly lower than that of Z250, and comparable to that of other tested composite resins. All tested composites had a FS higher than 80 MPa recommended by ISO-4049 for restorations under occlusal forces. Both CEF and CF showed significantly lower µSBS than the tested conventional composite resins.

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