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Original Article

Microshear bond strength of composite resins to enamel and porcelain substrates utilizing unfilled versus filled resins

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ABSTRACT

Background: Failures such as marginal discoloration and composite chipping are still the problems of tooth-colored restorations on the substrate of enamel and porcelain, which some of these problems are consequently as a result of failures in the bonding layer. Using filled resin has been recently introduced to increase the bond strength of this layer. The aim of this study was to compare the microshear bond strength (μ-SBS) of composite resins to enamel incubated in periods of 24 h and 9 months and porcelain with unfilled resin and flowable composites (filled resin).
Materials and Methods: In this *in vitro* study, two groups of 75 enamel samples with different storage times (24 h and 9 months) and a group of 75 porcelain samples were used. They were divided into 5 experimental groups of 15 samples in each. Composite cylinders in tygon tubes were bonded on the surface of acid-etched enamel and pretreated porcelain. Wave, Wave MV, Wave HV, Grandioflow and Margin Bond were used as bonding agents. The μ-SBS was measured at the speed of 1.0 mm/min. The bond strengths were analyzed with one-way analysis of variance (ANOVA) test followed by Tukey test. $P < 0.05$ was selected as the level of statistical significance in this study.
Results: The results showed that for enamel (24 h), the μ-SBS of the Wave MV and Wave HV groups were significantly lower than the Margin Bond group. Tukey test indicated the absence of a significant difference between the μ-SBS of the Wave group and the Margin Bond group. However, the μ-SBS of the Grandioflow group was significantly higher than the one for the Margin Bond as a bonding agent. In enamel (9 months), there was a significant difference between the Grandioflow and Margin Bond groups. Regarding bonding to the porcelain the one-way ANOVA test did not show a significant difference among the groups.
Conclusion: This study revealed that flowable composites (filled resins) can be used instead of unfilled resins in bonding composite resins to enamel and porcelain substrates.

Key Words: Composite resins, dental enamel, dental porcelain, shear strength

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INTRODUCTION

In today's society, people are very concerned about their aesthetic appearance. As part of that appearance, their teeth play an integral role. Therefore, tooth-colored restorations have gained more acceptances and have become the treatment of choice in dentistry today. As a result there has been an abundance of research in resin composite technology and adhesive dentistry.[1-3]

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Since the introduction of the acid-etch technique proposed in 1955 by Buonocore *et al.*,[4] many researchers have tried to improve the bonding strength between the resin material and substrate surfaces.[5] Bonding to the enamel surface is accomplished with unfilled resins that are applied to the acid-etched surface of the enamel, which has a high surface energy.[6] This also holds true for bonding to porcelain and metal.[5] The reason for using unfilled resins for enamel bonding is the adequate wettability of the acid-etched enamel surface, retention and bonding to the composite resin.[7] The bond strength of resin to acid-etched enamel surface utilizing phosphoric acid is about 18-22 Megapascal (MPa). Comparatively, the bond strength of resin to the acid-etched porcelain surface with hydrofluoric acid is more than 25 MPa.[8-10] The bonding strengths to each surface is strong enough to tolerate the stress caused by polymerization shrinkage of the resin, which is about 18 MPa.[11]

Researchers have tried to improve the bonding strength at the interface between composite resins and substrates; firstly, using composite resins with better mechanical properties and a higher degree of stiffness. Secondly, increasing the degree of conversion; using different types of activators. Thirdly, adding fillers with different percentages and different particle sizes to bonding agents.[12]

Adding fillers to bonding agents increases the viscosity of the material, which leaves a very thin layer of unpolymerized resin (O_2 inhibited layer).[13] In recent studies, it has been shown that increasing the percentage of fillers in bonding agents improves their mechanical properties.[14,15] Other benefits include

1. Increase in the bond strength
2. Increase in the resistance against crack propagation
3. Decrease in the polymerization shrinkage
4. Decrease in the linear coefficient of thermal expansion and
5. Decrease in the polymer matrix of the material, which leads to increasing of the modulus of elasticity.[16-21]

Although adding fillers increases the stiffness of the bonding agent, adding an excess of fillers increases the viscosity of the resin. This compromises the penetration of resin into porosities of the acid-etched enamel and dentin; hence, the optimum percentage of filler is crucial.[13]

Since flowable composites have different viscosities, due to the percentage of fillers and the size of filler particles, they are expected to have a better bond strength as compared with unfilled resins. The resultant enamel bond is possible because of its better physical properties.

The purpose of the following study was to compare the microshear bond strength (μ-SBS) of composite resin to enamel and porcelain with enamel bond (unfilled resin) and different flowable composites in terms of filler size and percentage of filler loading.

MATERIALS AND METHODS

Tooth preparation

In this *in vitro* study, 50 human third molar teeth that were extracted within 3 months were used. Those teeth that were carious, discolored or anatomically abnormal were excluded from the study. The teeth were stored in a 0.5% solution of chloramine-T for 1 week (4°C). After 1 week, each tooth was cut longitudinally into 3 mm sections using a diamond disk (D and Z, Darmstadt, Germany). These sections were made buccolingually and cut at a location 2 mm below the cemento-enamel junction. They were then polished with a sandpaper disk (up to #600-grit) under running water. Then, they were randomly divided into two sets of five groups each; so that after preparing the enamel samples, one set were stored for 24 h in an incubator (PECO-Model: PI-455G, Esfahan, Iran) (37°C) and the other set for 9 months under the same conditions.

Preparing the enamel samples

For this study, the polished surfaces of teeth were acid-etched for 20 s by 37% phosphoric acid (Super Etch, Southern Dental Industries [SDI], Australia), followed by 15 s of rinsing with water. This was followed by drying the surfaces with an oil-free air spray. In this step, a chalky appearance was an index of an acceptable acid-etched enamel surface. On each of these dried surfaces, according to their groups, a medium was applied as a bonding agent. These groups were as follows: Group 1; flowable composite (Wave, SDI, Australia), Group 2; flowable composite (Wave MV, SDI, Australia), Group 3; flowable composite (Wave HV, SDI, Australia), Group 4; flowable composite (Grandioflow, Voco, Germany), Group 5 (control); Unfilled resin (Margin Bond, Coltene, Switzerland). The composition and manufacturer of the materials, which were used in this study is presented the [Table 1].

The bonding agents were applied through a micro brush to the pretreated surfaces of substrates. Thinning of the bonding agent was accomplished with an oil-free air spray from 15 cm distance. After applying, 5 s were given to the bonding agent to spread over the high surface energy of the enamel substrate. The bonding agents were then cured in ramp curing way for 20 s with a light curing unit (Radii plus, SDI, Australia) with a light intensity of 1200 mW/cm^2. The light curing unit was holded in a right angle with a distance of 1 mm from the surface.

In the next step, tygon tubes (Tygon Norton Performance Plastic Co, Cleveland, OH, USA) with an internal diameter of 0.7 mm and height of 1 mm were used. Composite resin, (Ice, SDI, Australia) shade A2, was packed into the tubes which were then cured to the substrate with the same light curing unit for 40 s. After 1 h of being at room temperature (23°C), the tygon tubes were cut with a scalpel blade and removed from the substrates. A total of 75 enamel samples were stored in distilled water in an incubator (37°C) for 24 h, while another 75 enamel samples were stored similarly for 9 months.

Preparing porcelain samples

A total of 75 porcelain blocks having the dimensions of 2 mm × 3 mm × 4 mm were baked from VMK95 (Vident, Brea, CA, USA) under the same conditions in the laboratory. Porcelain surfaces were also polished with wet sandpaper (up to 600-grit). Porcelain blocks were also divided into five experimental groups randomly. The polished surfaces were acid-etched for 60 s with 9.6% of hydrofluoric acid (Porcelain Etch Kit, Ultradent, USA), rinsed for 30 s and air-dried utilizing oil-free air for 10 s. A silane coupling agent (Porcelain Etch kit, Ultradent, South Jordan, UT, USA) was then applied and given 10 s for evaporation. After pretreatment of all the porcelain blocks, the next steps were the same as for preparing specimens on an enamel substrate, which were;

1. Applying the specified bonding agent according to the group of porcelain block,
2. Oil-free air drying,
3. Light-curing for 20 s with the same light curing unit,
4. Placing of the tygon tubes,
5. Packing composite resin into the tygon tubes,
6. Light curing the composite resins,
7. Removal of the tygon tubes with a scalpel blade after 1 h of storage at room temperature (23°C) and
8. Storing the specimens in distilled water in an incubator (37°C) for 24 h.

Microshear bond test

The testing device available at Shahid Beheshti Dental school was for measuring microtensile forces (Microtensile tester, Bisco, USA). In order to change the design of this testing device from microtensile to microshear, one flat metal part was needed. The part was fabricated using the lost wax method and polished at completion. On the middle of this metal part, three metal cylinders having diameters of 0.7 mm and heights of 8, 9 and 10 mm, were laser soldered along a straight line. This part was attached with a cyanoacrylate adhesive (Mitreaple, Beta Kymia, Istanbul, Turkey) to the microtensile device, thus changing it into a microshear device [Figure 1].

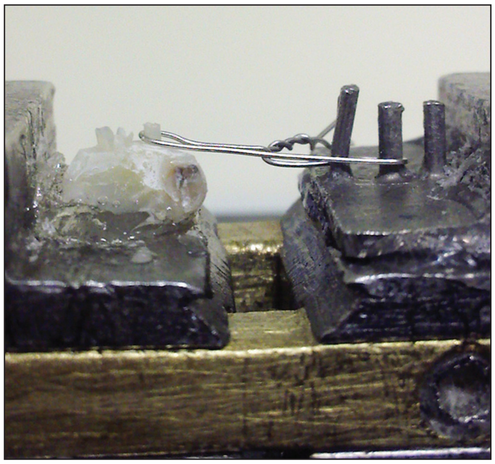

Figure 1: The part (in the right) which has been fabricated for changing the microtensile tester device in to microshear tester device

Table 1: Composition and manufacturer of materials used

Material	Containing	Manufacturer	Batch no.
Mitreapel	Ethyl-2-cyanoacrylate	Beta Kimya (Istanbul, Turkey)	8697464780092
Superetch	37% phosphoric acid	SDI (Australia)	0610149
Wave/A2	37% wt. multifunctional methacrylic ester 63% inorganic filler	SDI (Australia)	061194
Wave MV/A2	36% wt. multifunctional methacrylic ester 64% inorganic filler	SDI (Australia)	0610158
Wave HV/A2	34% wt. multifunctional methacrylic ester 66% inorganic filler	SDI (Australia)	0610153
Ice/A2	22.5% wt. multifunctional methacrylic ester (39% Vol.) 77.5% wt. inorganic filler (40 nm –1.5 µm) (61% Vol.)	SDI (Australia)	061144N
Porcelain Etch and silane	9% hydroflouoric acid 92% isopropranol, methacryloxy propyl trimethoxy silane	Ultradent products Inc. (Utah, USA)	B32 LW
Grandioflow/A2	Bis-GMA, UDMA, TEGDMA 80% inorganic filler	Voco (Cuxhaven, Germany)	741791
Margin bond	Bis-GMA, Bis-EMA, TEGDMA	Coltene (Altstatten, Switzerland)	LA542

SDI: Southern Dental Industries; TEGDMA: Triethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate; Bis-GMA: Bisphenol a glycol dimethacrylate; Bis-EMA: Ethoxylated bisphenol a glycol dimethacrylate

Study samples were also attached with the same cyanoacrylate adhesive on the other side of this device along with the metal cylinders on the other side. A thin wire with a diameter of 0.25 mm was looped around the composite resin cylinder base and on the other side around on the base of the metal cylinder. Microshear force with the crosshead speed of 1 mm/min was applied until a failure in bonding occurred. The amount of force that caused the failure in bonding was identified and converted to MPas using the formula "$F/\pi r^2$".[22] In this formula "r" is the radius of the cross section of cylinders and "F" is the amount of force that resulted in bonding failure. This study was carried out by the technique introduced by Shimada *et al*.[23] for measuring the μ-SBS. The data analysis was performed by SPSS 16 (SPSS for Windows; SPSS Inc., Chicago, IL, USA) and the Kolmogorov-Smirnov test was used for normal distribution. For statistical analysis, the One-way analysis of variance (ANOVA) test was used and once significant, a *post-hoc* Tukey honestly significance difference (HSD) test was conducted for individual comparisons. A *P* value <0.05 was selected as the level of statistical significance in this study.

RESULTS

The average μ-SBS of each experimental group for enamel (24 h) is shown in [Table 2], [Figure 2]. The bond strengths for enamel (9 months) are shown in [Table 3] and [Figure 3]. The bond strengths for Porcelain (24 h) are shown in [Table 4] and [Figure 4].

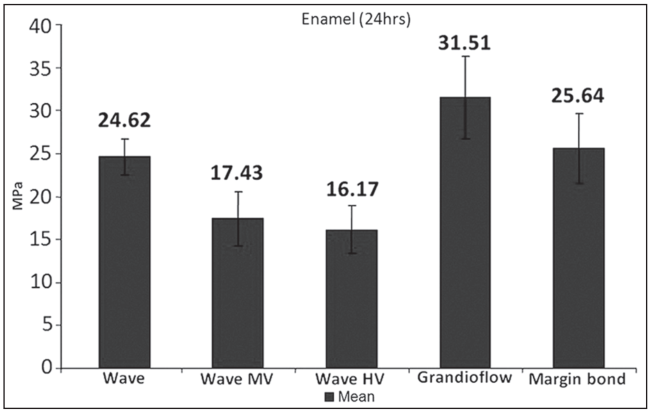

Figure 2: Mean microshear bond strengths for enamel samples which were stored in incubator for 24 h

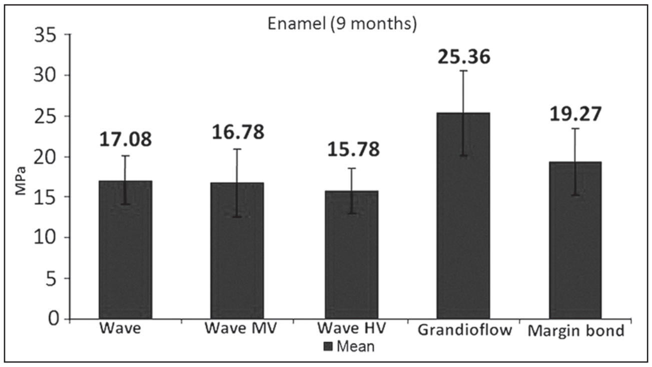

Figure 3: Mean microshear bond strengths for enamel samples which were stored in incubator for 9 months

Table 2: Micro shear bond strengths for enamel samples (24 h)

Bonding agent	*N*	Mean	Standard deviation	Variance	95% confidence interval		Minimum	Maximum
					Lowest value	Highest value		
Wave	15	24.62	1.45	2.12	23.44	25.8	20.27	28.59
Wave MV	15	17.43	1.76	3.12	15.7	19.16	13.25	23.65
Wave HV	15	16.17	1.67	2.8	14.62	17.72	12.73	22.09
Grandioflow	15	31.51	2.19	4.81	34.18	28.84	23.13	40.03
Margin bond	15	25.64	2.01	4.08	23.37	27.9	18.19	36.13

Table 3: Micro shear bond strengths for enamel samples (9 months)

Bonding agent	*N*	Mean	Standard deviation	Variance	95% confidence interval		Minimum	Maximum
					Lowest value	Highest value		
Wave	15	17.08	1.74	3.04	15.39	18.76	12.73	24.43
Wave MV	15	16.78	2.04	4.17	14.46	19.09	12.21	27.03
Wave HV	15	15.78	1.66	2.76	14.25	17.31	12.73	21.57
Grandioflow	15	25.36	2.28	5.22	22.46	28.25	19.94	34.05
Margin bond	15	19.27	2.03	4.14	16.97	21.56	14.98	27.55

Table 4: Micro shear bond strengths for porcelain samples

Bonding agent	*N*	Mean	Standard deviation	Variance	95% confidence interval		Minimum	Maximum
					Lowest value	Highest value		
Wave	15	25.51	1.52	2.33	24.21	26.80	19.75	28.59
Wave MV	15	24.1	1.65	2.73	22.58	25.61	18.19	28.07
Wave HV	15	23.08	2.56	6.57	19.44	26.72	15.59	39.77
Grandioflow	15	26.81	2.24	5.05	24.01	29.60	20.27	36.13
Margin bond	15	24.71	2.64	6.98	20.84	28.58	15.59	40.81

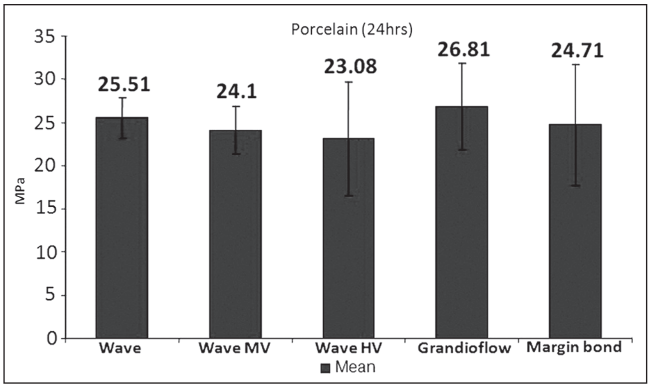

Figure 4: Mean microshear bond strengths for porcelain samples

The results of the one-way ANOVA showed significant differences among five experimental groups of enamel, which were stored for 24-h in an incubator. For individual comparisons, the Tukey HSD test was used. The result of this test showed no significant differences between the Wave and Margin Bond group ($P = 0.932$) and between Wave MV and Wave HV group ($P = 0.864$); however, the differences among other groups were significant ($P < 0.05$) [Table 5]. For groups, which enamel specimens were stored for 9 months in an incubator, the one-way ANOVA test showed significant differences among them. The result of the Tukey HSD test showed significant differences between Grandioflow and the Margin Bond group. There were no significant differences among other groups [Table 6]. For Porcelain groups, the one-way ANOVA test showed no significant differences among five experimental groups ($P > 0.05$).

Table 5: Results of *post-hoc* Tukey test HSD for enamel samples (24 h)

Relation	*P* value	Mean difference	95% confidence interval
Grandioflow > Margin bond	0.000*	5.871	2.269-9.472
Grandioflow > Wave	0.000*	6.890	3.288-10.491
Grandioflow > Wave MV	0.000*	14.082	10.481-17.684
Grandioflow > Wave HV	0.000*	15.340	11.738-18.941
Margin bond > Wave	0.932	1.018	−2.582-4.620
Margin bond > Wave MV	0.000*	8.211	4.609-11.812
Margin bond > Wave HV	0.000*	9.468	5.867-13.070
Wave > Wave MV	0.000*	7.192	3.591-10.794
Wave > Wave HV	0.000*	8.450	4.848-12.051
Wave MV > Wave HV	0.864	1.257	−2.344-4.858

*$P < 0.005$; HSD: Honestly significant difference

Table 6: Results of *post-hoc* Tukey test HSD for enamel samples (9 months)

Relation	*P* value	Mean difference	95% confidence interval
Grandioflow > Margin bond	0.001*	6.088	2.027-10.149
Grandioflow > Wave	0.000*	8.277	4.216-12.338
Grandioflow > Wave MV	0.000*	8.580	4.518-12.641
Grandioflow > Wave HV	0.000*	9.577	5.516-13.638
Margin bond > Wave	0.560	2.188	−1.872-6.249
Margin bond > Wave MV	0.430	2.491	−1.569-6.552
Margin bond > Wave HV	0.126	3.488	−0.572-7.549
Wave > Wave MV	1.000	0.302	−3.758-4.363
Wave > Wave HV	0.897	1.300	−2.761-5.361
Wave MV > Wave HV	0.959	0.997	−3.063-5.058

*$P < 0.005$; HSD: Honestly significant difference

DISCUSSION

One of the methods for improving the bond strength is to enhance the physical and mechanical properties of the adhesive layer. Adding fillers is one method of achieving this goal.[24-30] In this *in vitro* study, the μ-SBS of composite resins to enamel and porcelain, with different flowable composites as the bonding agents, were measured and compared with an unfilled resin as the control group. The flowable composites that were used in this study were Wave, Wave MV, Wave HV and Grandioflow. The first three flowable composites are the same in every aspect, except their percentage of filler contents and therefore their viscosities differ. The last flowable composite is filled with nano fillers [Table 1].

If the flowability of the composite resin is enough for adequate wetting of the surface of the pre-treated substrate, it can be used without any bonding agents. Most types of luting cements and flowable composites are flowable enough to penetrate into the enamel tags of acid-etched porcelain's porosities.[31] The other advantage of flowable composites over unfilled resins is the variety of the shades that they have. They have almost covered all the shades in the Vita Shade Guide;[31] Furthermore, because of the existence of the fillers, the polymerization shrinkage of flowable composites is less than in unfilled resins.[5,25] These characteristics prevent the formation of white lines at the cavosurface margin of composite resin fillings.

The μ-SBS test was selected for measuring the bond strength because of its simplicity and reliability.[23]

Enamel (24 h)

Based on the results of this study, of those groups of enamel which were stored for 24 h in an incubator, Grandioflow showed the highest bond strength, when compared with the others. This difference was statistically significant ($P < 0.05$). Margin Bond and the Wave groups came in second. This means that the bond strength of these two groups did not show any significant difference compared with each other ($P = 0.932$). The weakest bond strength was for Wave MV and Wave HV groups. There was also an absence of a significant difference between these two groups ($P = 0.864$).

This data agrees with other similar studies in this field.[24,25,27-30,32,33] In other words, using flowable composites instead of unfilled resins can make an acceptable bond between the composite resin and enamel. However, the viscosity of the flowable composite has to be considered regarding its wettability. Tecco *et al*.,[30] Ryou *et al*.,[29] Frankenberger *et al*.,[27] and D'Attilio *et al*.[25] also have studied this phenomenon. When the viscosity of flowable composite increases, the bond strength decreases because the act of penetration of the resin into the acid-etched enamel tags is compromised. This is the reason that Wave HV and Wave MV groups which have high and medium viscosity, respectively,[34] have the weakest bond strengths. As it is shown in [Figure 5], following Margin Bond (control), which is an unfilled resin material, Grandioflow is the least viscous and most wettable bonding agent among all other flowable composites; therefore, this characteristic identifies this material with the highest bond strength when compared with other flowable composites. Although Grandioflow is more viscous than Margin Bond, it created a stronger bond as compared with the Margin Bond group. Faltermeier *et al*. showed that 70% filled urethane dimethacrylate resin will create the highest bond strength between orthodontic brackets and enamel compared with a 30% and 50% filled resin.[26] Since Grandioflow has 80% inorganic fillers, this study agrees with their results. Furthermore, Grandioflow is a nanohybrid flowable composite, which means that the space between microfillers is filled with nanofillers (10-100 nm). As in filled dentin bonding agents, the size of fillers is the most important factor in penetrating into the dentinal tubules.[35] The size of fillers is also one of the important factors in enamel bonding. Considering the size of enamel tags (macrotags: 4 μm, microtags: 0.05 μm),[36] infiltration of micro fillers into macrotags and nanofillers into microtags will enhance the bond strength between composite resins and enamel. The existence of fillers which improves the mechanical and physical properties of the bonding agent will also enhance the bond strength. Bishara *et al*. mentioned that Grandioflow has a high wear resistance and low marginal leakage because of nanofillers.[24] The presence of nanofillers created higher bond strengths compared with all other experimental groups. Uysal *et al*. studied the effects of the bond strength of orthodontic brackets to enamel with three kinds of flowable composite in comparison with a conventional orthodontic adhesive.[37] Their study showed that using flowable composites as a bonding agent leads to decreased bond strength compared with a conventional orthodontic adhesive. In their study, the viscosity of flowable composites and light curing over the metal orthodontic bracket might have resulted in unacceptable bond strength.

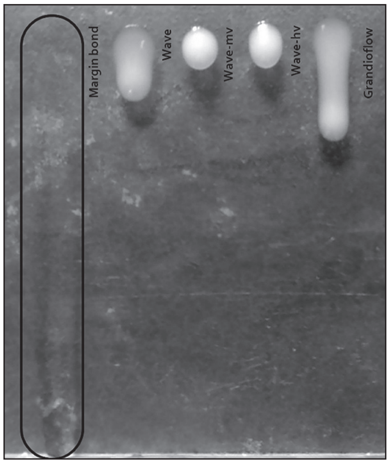

Figure 5: Flow of bonding agents which were used in this study at 90° angle for 5 min

Enamel (9 Months)

For those groups that were stored for 9 months in an incubator for evaluating the longevity of bond strengths, despite water absorption and hydrolysis of the bonding layer, the Grandioflow group had the highest bond strength. This finding was statistically significant ($P < 0.05$). According to the paired *t*-test that was performed for comparing each group with the same groups that were stored for 24 h, the mean μ-SBS for each of the five experimental groups was decreased significantly. These results agree with other similar studies.[38-41] De Munck *et al*.[38] and Jaberi-ansari and Sadr,[40] in their studies assessed the longevity of bond strengths of composite resins and their results also agree with this study.

Porcelain

As it is shown in Figure 4, although the μ-SBS of the Grandioflow group is the highest among all other groups, the one-way ANOVA test showed no significant difference among the five experimental groups. Therefore, flowable composites can be used as a bonding agent for repairing porcelains with composite resins. In a study by Barceleiro Mde *et al*. showed that using a flowable composite can be an alternative for bonding porcelain laminate veneers instead of using a dual cure resin cement.[42] Since the surface tension and free surface energy of porcelain after etching with hydrofluoric acid is higher than the surface tension of enamel after being etched with phosphoric acid,[43-45] and also the porosities of acid etched porcelain are larger,[46] the viscous flowable composites are also able to penetrate into the porosities of the acid etched porcelain surface. Therefore, the role of viscosity of bonding agents for porcelain is less important. Furthermore, as it is stated in other studies, most of the failure modes between porcelain and composite resins are cohesive. The reason is that the acid-etching technique in porcelain reduces the cohesive strength of the surface of the porcelain.[47] This means that the bond strength between composite and porcelain is high enough to tolerate the forces, so the load will fracture the porcelain itself.

Yassini and Tabari in their study showed that using dentin bonding agents as filled resins for bonding composite resins to porcelain have a higher bond strength when compared to flowable composites.[47] The reason for this result was that in their study, a water based silane coupling agent was used in the bonding procedure. After the porcelain surface dried, some amount of water remained on the surface. This reduced the free surface energy of the porcelain; hence, the viscosity of the bonding agent played a crucial role in bonding. In this study, an alcohol based silane coupling agent was used, in order to avoid reducing the free surface energy of the porcelain surface and having optimum bond strength.

CONCLUSION

This study revealed that, with respect to the viscosity of bonding agents, flowable composites can be used as bonding agents in lieu of unfilled resins. Grandioflow, a nanofilled flowable composite, created stronger bonds than the unfilled resin, Margin Bond. Flowable composites can be substituted for unfilled resin without any significant change in bond strength to the porcelain substrate.

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