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Original Article

Effects of surface treatments of conventional glass-ionomer on shear bond strength to giomer

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ABSTRACT

Background: An appropriate bond between glass-ionomer and the superficial resin materials is very important for the success of sandwich technique. The aim of the present *in vitro* study was to evaluate the effect of three surface treatments of conventional glass-ionomer on its shear bond strength to giomer.
Materials and Methods: Sixty cylindrical specimens of a conventional glass-ionomer (GC Fuji II) were prepared and randomly divided into three groups ($n = 20$). The specimens in groups 1 and 2 were treated with total-etch adhesive resin (Single Bond) along with acid etching, and self-etch adhesive resin (FL-Bond II) on the set glass-ionomer, respectively. Specimens in group 3 were treated with self-etch adhesive resin (FL-Bond II) before initial setting of the glass-ionomer was complete. Then a giomer restorative (Beautifil II) was added to the specimens. Subsequent to thermocycling, the specimens were subjected to shear bond strength test. Failure modes were evaluated under a stereomicroscope. Data were analyzed by one-way analysis of variance and a *post hoc* Tukey test at a significance level of $P < 0.05$.
Results: There were statistically significant differences in bond strengths between the groups ($P < 0.0005$). Differences in bond strengths between group 2 and other groups were significant ($P < 0.0005$) while the differences between groups 1 and 3 were not significant. Failures were predominantly of the cohesive type in all the groups.
Conclusion: Based on the results of this study, the use of self-etch adhesive resin (FL-Bond II) on the set glass-ionomer yielded the highest bond strength in the glass-ionomer/giomer sandwich technique.

Key Words: Bond strength, giomer, glass-ionomer cement, sandwich technique, surface treatments

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INTRODUCTION

A sandwich or laminate technique is one of the methods proposed for composite resin restorations, which was introduced for the first time by McLean, *et al*. in 1985. The basic idea behind this technique is to use two different restorative materials for a single restorative procedure so that the maximum physico-mechanical and esthetic properties of these two materials can be exploited simultaneously.[1,2] Generally, the first component is a layer of conventional or resin-modified glass-ionomer cement which has drawn attention due to its capacity to form an inherent bond with tooth structures and the resultant better seal and decrease in microleakage (particularly in dentinal walls); it can also release fluoride and decrease the odds of carious lesions.[1,3] The second component is a layer of composite materials (including composite resin, compomer, and ormocer), which is used to compensate for the limitations and disadvantages of glass-ionomer cements, including weak mechanical properties and inappropriate esthetic appearance.[1] It

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has been reported that the combination of these two materials have different property results in the clinical success of restorations.[1,3]

The success of the laminate technique depends on the strength of the bond between the glass-ionomer and the resin composite materials in addition to the strength of the bond between the glass-ionomer cement and dentin.[3] However, there is a relatively weak bond strength between conventional glass-ionomers and composite resin materials, mainly because of the lack of a chemical bond between these two materials and the low cohesive strength of glass-ionomer.[3-5] Several studies have evaluated different surface preparation techniques for glass-ionomers, such as the use of different bonding systems and surface etching procedures in order to increase the bond strength between these two materials in the laminate technique.[1,3,4,6,7] Etching the surface with phosphoric acid has yielded different results. In a study carried out by Sheth, *et al*. etching the surface of glass-ionomer had no effect on bond strength increase;[8] however, an increase in bond strength after acid etching has been reported in another study.[9] It has also been reported that premature etching of glass-ionomer cement (before its initial setting reaction) and failure to use an adhesive resin between the glass-ionomer cement and composite resin increases the odds of restoration failure.[10] In another study, it was demonstrated that the use of a self-etch adhesive system on half-set glass-ionomer cement (before its initial setting) increases the bond strength between the glass-ionomer cement and composite resin more than that observed with the use of total-etch systems (its application after the initial setting of the cement). It has also been reported that the use of a glass-ionomer-based adhesive system applied after the cement's initial setting improves the bond strength between the glass-ionomer cement and composite resin compared to that with the use of total-etch adhesive systems under similar conditions.[3]

A new group of composite resin materials, giomers, have been introduced in less than a decade, which consist of reacted glass-ionomer fillers in a resin matrix; they are used in cavities in a manner similar to composite resins with the application of an adhesive system. In addition to appropriate esthetic results, easy polishing, fluoride recharging potential and strength,[11,12] these materials release fluoride which may enhance their antibacterial effects.[13,14] Clinical success of giomer restorations has been demonstrated in several studies.[15,16] However, no studies to date have evaluated the bond strength between glass-ionomers and giomers; therefore, the aim of the present study was to evaluate the effect of three surface preparation methods on the shear bond strength of giomer to different surface treated conventional glass-ionomer.

MATERIALS AND METHODS

Sixty cylindrical specimens were used in the present *in vitro* study. In order to prepare the samples, a plastic mold (with an inner diameter of 6 mm and a height of 4 mm) was placed on a glass slab. Then conventional glass-ionomer (GC Fuji II, GC Corporation, Tokyo, Japan) was packed into the plastic mold after mixing according to the manufacturer's instructions. Another glass slide was used on the other side of the mold to make the free surface of the conventional glass-ionomer smooth. The samples were not finished in order to simulate the clinical situation.[3] Then the samples were randomly divided into three groups of 20. According to the manufacturer, the initial setting of GC Fuji II conventional glass-ionomer lasts in 5 minutes and a half, and the net setting time is 2 minutes and a half. Three groups were compared; a total-etch adhesive resin or a self-etch giomer-based adhesive resin on set glass-ionomer; or the self-etch giomer-based adhesive resin on the unset glass-ionomer (before completion of the initial setting).

In group 1 the surfaces of the specimens were etched with 37% phosphoric acid (3M ESPE Dental Products, St. Paul, MN, USA) for 15 seconds after 5 minutes and 30 seconds from the mixing procedure; the initial setting reaction was confirmed with the use of a sharp dental explorer.[3] It should be pointed out that a complete setting of conventional glass-ionomer takes 24-72 hours. After surface etching, a Single Bond total-etch adhesive system (3M ESPE Dental Products, St. Paul, MN, USA) was applied according to the manufacturer's instructions and light cured [Table 1]. A second transparent mold, with a diameter of 3 mm and a height of 2 mm, was placed on the conventional glass-ionomer specimen and a giomer restorative, Beautifil II A3-shade (Shofu Dental Corporation, Osaka, Japan), was packed into the transparent mold and cured using a halogen light-curing unit (Astralis 7; Ivoclar Vivadent, FL-9494 Schaan, Liechtenstein). The light-directing

Table 1: Chemical composition and application mode of adhesives used

Adhesive	Composition	Application mode
Single Bond (3M ESPE) Two-step etch and rinse	Etchant: 37% phosphoric acid gel Adhesive: HEMA*, Bis-GMA†, ethanol, water, polyalkenoic acid copolymer	Apply the etchant for 15 seconds; rinse for 10 seconds; apply two coats of adhesive; gently air-dry the surface for 5 seconds and light cure for 10 seconds
FL-Bond II (Shofu Dental Corporation)	FL-Bond primer: Carboxylic acid monomer, phosphonic acid monomer, water, solvent, initiator	Apply and leave for 10 seconds and air dry the surface for 5 seconds
Two-step giomer-based self-etch	FL-Bond Bonding: S-PRG filler‡, UDMA§, TEGDMA‖, HEMA*, initiator	Apply an even layer and light cure for 10 seconds

*[2]-hydroxyethyl methacrylate; †Bisphenol-glycidyl methacrylate; ‡Surface reaction type prereacted glass-ionomer filler; §urethane dimethacrylate; ‖Triethyleneglycol dimethacrylate

probe had a diameter of 8 mm and directed a light ray at an intensity of 700 mW/cm^2 perpendicular to the surface, barely touching the surface, for 40 seconds. A light intensity of 700 mW/cm^2 was confirmed using a radiometer before the start of each experimental session. After transparent mold removal, the specimens were cured for another 20 seconds from each direction. Then the samples were kept in humidity chamber at 37ºC for 1 hour before being immersed in distilled water at 37ºC for the next 23 hours. In the next stage, a 500-cycle thermocycling procedure was carried out at 5ºC/55ºC ± 5ºC with a dwell time of 30 seconds and a transfer time of 10 seconds.

In group 2, the procedures were similar to those in group 1 except that FL-Bond II (Shofu Dental Corporation, Osaka, Japan) self-etch adhesive resin was used on set glass-ionomer surface according to the manufacturer's instructions and light cured [Table 1], without acid etching.

In group 3, all the procedures were similar to those in group 2 except that FL-Bond II adhesive was applied according to the manufacturer's instructions right after the initial setting and before hardening of glass-ionomer (2 minutes and a half after the initiation of mixing, which is equal to the cement's net setting time). It should be pointed out that during that time the surface hardness of the cement was sufficient to place the second mold without damaging the cement.

In order to measure the shear bond strength, the samples were mounted in cold-cured acrylic resin from the glass-ionomer side and the shear bond strength values of the samples were measured in Newton using a universal testing machine (H5K-S model, Hounsfield Test Equipment, Surrey, UK) at a crosshead speed of 1 mm/min using a 0.5 mm-wide chisel. Then the shear bond strength values were calculated in MPa by dividing the force (in Newton) by the surface area (mm^2) of the samples. Data were analyzed by one-way analysis of variance (ANOVA) and pairwise comparisons were made by a Tukey test using SPSS/win.15. Statistical significance was set at $P < 0.05$.

In all the groups, the failure modes were determined under a stereomicroscope (SMZ1500, Nikon, Tokyo, Japan) at ×20. The failure modes were classified as follows:[3]

Adhesive failure: Failure at giomer–glass–ionomer cement interface.

Cohesive failure: Failure within giomer or glass-ionomer cement.

Mixed failure: A combination of the two above-mentioned modes.

In addition, to evaluate the interface between the conventional glass-ionomer and giomer in the groups under study, two additional specimens were prepared in each group and subsequent to longitudinal sectioning, they were evaluated under an scanning electron microscope [SEM] (Tescan, Vega II XMU, Brno, Czech Republic) [Figure 1].

RESULTS

Shear bond strengths in MPa (means and standard deviations) for the study groups are represented in Table 2. There were statistically significant differences in shear bond strength values between the study groups ($P < 0.0005$). Pairwise comparisons by a Tukey test revealed that there were statistically significant differences in shear bond strengths between group 2 and the two other groups ($P < 0.0005$), whereas the bond strength difference between groups 1 and 3 was not statistically significant ($P = 0.609$).

Failure modes of the study groups are shown in Table 3. Failures were predominantly of the cohesive type in all the groups. Moreover, all the cohesive failures were within glass-ionomer cement and there were no cohesive failures inside the giomer.

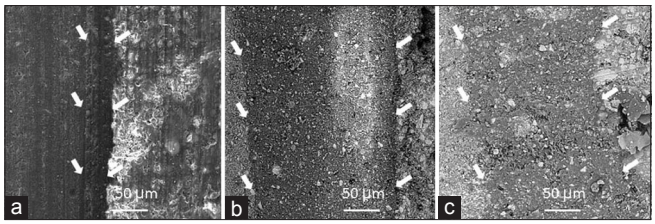

Figure 1: Scanning electron micrographs of conventional glass-ionomer and giomer interface in the study groups (Mag ×500). Arrows indicate margins of adhesive resin between glass-ionomer and giomer. In the three scanning electron micrographs, the materials which are placed on the right and left sides of the adhesive resin indicate glass-ionomer and giomer, respectively

Table 2: Means and standard deviations (SD) of the shear bond strengths (MPa) measured in study groups

Groups	Surface treatment	Mean±SD	(*n*)
1	Total-etch+set glass-ionomer	3.24±0.45	20
2	Self-etch+set glass-ionomer	6.20±0.35	20
3	Self-etch+unset glass-ionomer	3.12±0.30	20

Table 3: Failure modes of the study groups

Groups	Surface treatment	Adhesive	Cohesive	Mixed	(*n*)
1	Total-etch+set glass-ionomer	5	10	5	20
2	Self-etch+set glass-ionomer	5	13	2	20
3	Self-etch+unset glass-ionomer	3	11	6	20

SEM photomicrographs of the glass-ionomer and giomer interface in the study groups are represented in Figure 1. In the SEM photomicrograph in group 1 [Figure 1a] the hybrid zone (the area of adhesive resin penetration into glass-ionomer) is almost homogeneous but has irregular borders on the glass-ionomer cement side. In the SEM photomicrograph in group 2 [Figure 1b] the hybrid zone is regular and homogenous and in the SEM photomicrograph in group 3 [Figure 1c] the hybrid zone is irregular and non-homogenous. Moreover, the use of self-etch adhesive resin (FL-Bond II) in groups 2 and 3 resulted in the thicker hybrid zone than total-etch adhesive resin (Single Bond) in group 1.

DISCUSSION

An appropriate bond between glass-ionomer and the superficial resin materials is very important for the success of the sandwich technique.[3] In the present study shear bond strength of conventional glass-ionomer to giomer was evaluated using three different surface preparations for glass-ionomer (use of self-etch adhesive on glass-ionomer with or without complete initial setting reaction and use of total-etch adhesive on glass-ionomer after completion of the initial setting reaction). In designing the present study, the use of total-etch adhesive on glass-ionomer without the completion of the initial setting reaction was not considered, because in total-etch adhesives, rinsing after etching and contamination with moisture before completion of the initial setting reaction of glass-ionomer influences the surface integrity of the cement.[3]

The results of the present study showed that shear bond strength of glass-ionomer to giomer depends on surface preparation. In this context, in the group in which glass-ionomer was set and self-etch adhesive was used, the highest shear bond strength was recorded compared to two other groups, which is consistent with the results of a study carried out by Gopikrishna, *et al*. on glass-ionomer-based adhesives; in the case of complete setting reaction the bond strength was higher than that in incomplete setting reaction.[3] Contrary to the results of the present study, Knight, *et al*. reported no statistically significant differences in the bond strength before and after initial setting of glass-ionomer in the co-cure technique, where conventional glass-ionomer, resin-modified glass-ionomer (RMGI), and composite resin were placed in consecutive layers before light-curing procedure.[17] The differences in the results of that study and the present study might be attributed to different etching times in the two studies. The etching time was 15 seconds in the present study, while in the study done by Knight, *et al*., it was only 5 seconds. It appears that longer etching time paves the way for greater destruction of glass-ionomer surface by the acid and decreases the bond strength. In addition, a different technique was used in the above-mentioned study, i.e., a layer of RMGI was used over conventional glass-ionomer before placement of composite resin and the curing process was carried out for RMGI and composite resin simultaneously.[17]

It is well-known that the pH of glass ionomer cements is strongly acidic upon mixing, and it will increase with time, with the most rapid increase during the first 5-10 minutes of setting, regardless of the composition of the glass ionomer material.[18] In comparison of groups 2 and 3, it appears that the acidic pH of unset glass-ionomer prevented complete polymerization of the giomer-based self-etch adhesive and therefore decrease the bond strength.[19,20] In addition, this

acidic pH might have an adverse effect on the polymerization of giomer itself. In a study carried out by Mohamed-Tahir, *et al*. it was demonstrated that an acidic environment decreased microhardness of giomer.[21] Another study has shown that surface hardness of composite resin placed on polyalkenoate glass (set for 4 minutes) significantly decreased.[22] Moreover, SEM observation of the interface in group 3 revealed an inhomogeneous structure and occasionally breakdown of the glass-ionomer surface, which may have affected the bond strength. Polyacrylic acid prevents polymerization of composite resin and results in composite resin softening;[22] apparently this effect was intensified in incompletely set glass-ionomer.

In comparison of groups 1 and 2, it appears that etching the glass-ionomer surface immediately after the initial setting in group 1 in the total-etch system resulted in the destruction of cement surface, crack formation and decrease in bond strength. This speculation was confirmed by the SEM micrographs of the interface. The irregular interface in group 1 may be an indication of damage and weakening of the glass ionomer surface rather than improved micromechanical interlocking, when compared to group 2 which was treated by a comparatively mild acid, i.e., the self-etching primer.

Several studies have demonstrated a decrease in bond strength of glass-ionomer to composite resin as a result of etching the glass-ionomer surface.[9,23,24] It has been reported that etching during the initial setting reaction of glass-ionomer leads to dissolution of weak calcium-polyacrylate rings, with the resultant deterioration of its physical properties.[3] In the total-etch procedure, the bond between giomer and glass-ionomer seems to be completely micromechanical.[3] However, another reason for a higher bond strength in the self-etch group compared to the total-etch group is the fact that the acidic monomers in the self-etch primer can chemically bond to the calcium in glass-ionomer and increase bond strength.[3,11] In addition, a more appropriate compatibility of giomer with the giomer-based self-etch adhesive might be another reason for a higher bond strength in group 2; it has been suggested that the use of adhesives compatible with resin-based restorative materials can decrease deleterious chemical interferences.[25] Moreover in the self-etch adhesive group, resin penetration occurs simultaneous with the etching process and it is probable that the discrepancy between these two processes is eliminated or minimized.[3,4] Another factor that needs to be taken into account is the difference in composition and mechanical properties of the two adhesives; FL-Bond II is a two-step self-etching adhesive with filler particles while Single Bond is a mixture of hydrophilic monomers and solvents that contains no fillers. It has been suggested that the two-step self-etching adhesives that incorporate a hydrophobic resin as a separate bonding agent may have enhance mechanical properties compared to simplified adhesives. Moreover, apart from the composition of resin matrix, addition of filler particles enhances the mechanical strength of the bonding layer and contributes to bond strength.[26] In the present study, no differences were observed in bond strength between groups 1 and 3, and bond strength in both groups was significantly less than that in group 2.

Regarding failure mode in the present study, the majority of failures were of the cohesive type in glass-ionomer, which is consistent with previous studies.[1,27] This phenomenon might be attributed to lower mechanical properties of glass ionomer cements when compared to resin-based materials and the presence of numerous air inclusion bodies inside glass-ionomer, which act as stress concentration points and probably increase the odds of cohesive failure.[10] A disadvantage of sandwich technique is the absence of a chemical bond between conventional glass-ionomer and superficial resin materials; therefore, researchers have focused on the establishment of a chemical bond between them. Considering the results of the present study, use of giomer-based self-etch systems not only decreases the time needed for the clinical application but also can result in the establishment of a chemical bond between giomer and conventional glass-ionomer.

CONCLUSION

Taking into account the limitation of this *in vitro* study, etching the surface of set glass-ionomer with a total-etch system or placement of self-etch adhesive on the surface of glass-ionomer with incomplete initial setting compromised bonding of giomer to glass-ionomer.

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