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RESEARCH ARTICLE

Modified Microtensile Bond Strength of Glass Ionomer to Composite Resin Using Universal Adhesive in Self-etch and Total-etch Modes

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Abstract:

Background:

Glass Ionomer Cements (GICs) are frequently used as base or liner before the application of restorative materials. The success of this approach depends on the bond strength of GICs to composite resin.

Objectives:

This study to assess the modified microtensile bond strength of glass ionomer to composite resin using universal adhesive in self-etch and total-etch modes.

Methods:

Samples were fabricated of resin-modified GIC (RMGIC) and conventional GIC (CGIC) (6 x 1 x 1 mm), and were randomly divided into 8 groups. Clearfil SE Bond and G-Premio universal adhesive in self-etch and total-etch modes were used according to the manufacturers' instructions. Z250 composite was applied over the GIC (12 x 1 x 1 mm), and light-cured. The microtensile bond strength was measured using a universal testing machine. The samples in each group were evaluated under an electron microscope to determine the mode of failure. Data were analyzed using one-way ANOVA and Tukey's test.

Results:

The microtensile bond strength of RMGI used with Clearfil SE Bond was significantly higher than that of other groups (6.57±1.15 MPa) (P<0.05). The maximum and minimum microtensile bond strength values of CGIC after applying the bonding agents were recorded after using G-Premio total-etch mode (1.34±0.77 MPa) and SE Bond in total-etch mode (1.18±0.79 MPa), respectively.

Conclusion:

Application of G-Premio in both modes did not show any significant different bond strength in both glass ionomers. The bond strength of RMGIC was higher than that of CGIC, and the maximum bond strength of RMGI was achieved by the use of SE Bond.

Keywords: Composite resin, Glass ionomer cement, Dentin bonding agents, Tensile strength, Glass ionomer cements, Bond strength.

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1. INTRODUCTION

With the increasing demand for esthetic dental treatments, a wide range of esthetic dental materials in two major groups

of glass ionomer cements (GICs) and composite resins are now available in the market. Composite resins are among the most commonly used esthetic restorative materials with favorable properties such as optimal elastic modulus, flexural strength, hardness, and wear resistance [1,2]. However, satisfactory bonding of composite resin to dentin and cementum in deep cervical margins with optimal marginal seal is questionable due to the polymerization shrinkage stress and incomplete

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penetration of the bonding agent [1 - 3]. GICs have optimal properties such as chemical bonding to the moist tooth structure, fluoride release potential, coefficient of thermal expansion similar to that of tooth structure, optimal biocompatibility, and antibacterial and cariostatic activities [1, 4].

Application of GICs in combination with composite resins, known as the sandwich technique, is an effective strategy to benefit from the favorable properties of both materials in one restoration [5 - 7]. Assessment of the mechanical behavior of materials in large class I composite restorations revealed a reduction in stress level at the dentin/restoration interface by the use of this technique [8]. However, based on clinical evaluations, the application of GICs may weaken the restoration and increase the risk of fracture of composite restorations [9].

Nonetheless, GICs are still the best substitute for replacement of the lost dentin according to the biomimetic principles [10], and the sandwich technique is a commonly used restoration technique in the clinical setting. Additionally, this technique has been proposed for the restoration of deep proximal caries extending beyond the cemento-enamel junction, to avoid surgical crown lengthening [2]. However, one critical point in the long-term success of the sandwich technique is adequate bond strength between the GIC and composite resin, which is imperative for optimal stress transmission, retention, durability, and sealing [2,11].

Recently, universal adhesives were introduced to the market, which are capable of bonding to various substrates. Universal adhesives contain acidic monomers. They can be applied in both self-etch and etch-and-rinse modes [2]. A search of the literature by the authors yielded limited studies on the bond strength of GICs to composite resin mediated by the use of universal adhesives [2, 4, 12].

Several methods can be used to measure the bond strength between different restorative materials. These methods can be static or dynamic. Shear, microshear, tensile, and microtensile tests are commonly used for this purpose. The microtensile test has some advantages over the others due to the small size of the critical-size defects, more even stress distribution, higher reliability, and capability for assessment of irregular specimens [13 - 15]. However, the microtensile test has some drawbacks as well such as the induction of micro-cracks in the samples as a result of sectioning, higher technical demands, and underestimating the bond strength [15]. Sano *et al.* [16] discussed that pre-test failure is an important problem when using an adhesive with low microtensile bond strength. Thus, we designed a modified microtensile bond strength test for the evaluation of bond strength without sectioning the samples in this study. Since the oral environment is a dynamic environment, the durability of bonding is clinically important. Therefore, this study aimed to assess the modified microtensile bond strength of GICs to composite resin using a universal adhesive in self-etch and total-etch modes.

2. MATERIALS AND METHODOLOGY

Table 1 lists the materials used in this study. The sample size was calculated to be 10 in each group according to a study

by Kavian *et al.* [1] using advanced repeated measures ANOVA power analysis, considering $\alpha=0.05$, $\beta=0.2$, effect size of 0.51, and standard deviation of 2.5.

Table 1. Materials used in this study.

Composition	Manufacturer	Material
Acetone (25–50%), 2-hydroxy-1,3-dimethacryloxypropane (10–20%), methacryloyloxydecyl dihydrogen phosphate (5–10%), 2,2-ethylenedioxydiethyl dimethacrylate (1–5%), diphenyl(2,4,6-trimethylbenzoyl)-phosphine oxide (1–5%), 2,6-di-tert-butyl-p-cresol (<0.5%).	GC Corporation, Tokyo, Japan	G-Premio BOND
Powder: Fluoroaluminosilicate glass Liquid: Acrylic acid, maleic acid, HEMA, water, camphorquinone	GC Corporation, Tokyo, Japan	Fuji II LC
Polyacrylic acid 39% and tartaric acid 11%	GC Corporation, Tokyo, Japan	Fuji IX
Matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA; Filler: Zirconia/silica (averagely 01.0-5.3 μm)	GC Corporation, Tokyo, Japan	Z250
Primer: Water, MDP, HEMA, CQ, DET, hydrophilic DMA Bond: MDP, bis-GMA, HEMA, hydrophobic DMA, CQ, DET, silanated colloidal silica	Kuraray, US	Clearfil SE Bond

Forty specimens measuring 6 x 1 x 1 mm were fabricated of GICs using plexiglass molds according to the manufacturers' instructions. The conventional GIC (CGIC) samples were removed from the molds after 24 hours while the resin-modified GIC (RMGIC) samples were light-cured for 40 s using a light-curing unit (WoodPecker Medical Instruments, Guilin, China) with a light intensity of 800 mW/cm². The output of the device was periodically checked using a radiometer (DigiRate, Monitex, Taiwan). A total of 80 GIC samples were fabricated and randomly divided into 8 groups. RMGIC was used in groups 1 to 4 and CGIC was used in groups 5 to 8 as follows:

Group 1: RMGI, group 2: RMGI and ClearfilSE Bond, group 3: RMGI and G-Premio Bond (self-etch mode), group 4: RMGI and G-Premio Bond (total-etch mode), group 5: CGIC, group 6: CGIC and ClearfilSE Bond, group 7: CGIC and G-Premio Bond (self-etch mode) and group 8: CGIC and G-Premio Bond (Total-etch mode)

In groups 2 and 6, Clearfil SE Bond was applied on the fabricated GIC samples according to the manufacturer's instructions and after 20 s, it was thinned and dried with oil-free gentle air spray. Curing was performed for 10 s. Z250 composite resin was applied to the samples and light-cured for 40 s.

In groups 3 and 7, 37% phosphoric acid (GC Corporation, Tokyo, Japan) was applied to GIC samples for 10 s. They were then rinsed for 20 seconds dried with oil-free air spray. G-Premio Bond bonding agent was applied on the surface. After 10 s, it was dried with maximum air pressure for 5 sand light-cured for 10 s. The rest of the procedure was the same as that

in groups 2 and 6.

In groups 4 and 8, one layer of G-Premio Bond universal adhesive was applied on GIC samples (self-etch mode). After 10 s, it was dried with maximum air pressure for 5 sand cured for 10 s using a light-curing unit with a light intensity of 800 mW/cm² according to the manufacturer’s instructions. The rest of the procedure was the same as that in groups 2 and 6.

In groups 1 and 5 (control groups), GICs were used with no bonding agent.

Next, the samples were subjected to 3000 thermal cycles between 5±2°C and 55±2°C with a dwell time of 20 sand a transfer time of 5 to 10 s. Thermal cycles between 3000 and 100,000 cycles have been recommended, and it has been reported that 10,000 thermal cycles correspond to one year of clinical service [17]. Therefore, the samples underwent 3000 thermal cycles in this study in order to simulate 3 moths of clinical service in the oral environment. For microtensile bond strength test, the samples were fixed to the mold of the universal testing machine (Santam, Iran) with cyanoacrylate glue and subjected to 0.5 N load at a crosshead speed of 0.5 to 1 mm/min. The microtensile bond strength was reported in megapascals (MPa).

The samples were chosen from each group for further assessment of the interface of GIC and composite resin. The samples were gold-coated and evaluated under a scanning electron microscope (F40 FEI, Nova, USA). The samples were inspected under a relative vacuum to 10⁻⁷. Images were obtained with 1.4-1.8 nm resolution at x150 magnification. The mode of failure was categorized as adhesive (at the interface of GIC-composite), cohesive (within the GIC), or mixed (a combination of adhesive and cohesive failures).

Data were analyzed using SPSS version 25 (SPSS Inc., IL, USA). The microtensile bond strength of the groups was compared using ANOVA. Pairwise comparisons were carried out using the Tukey’s test. The level of significance was set at 0.05.

3. RESULTS

Table 2 shows the microtensile bond strength of the groups. Maximum microtensile bond strength was noted in group 2, and the minimum value was noted in group 5 (control). The microtensile bond strength of the groups in descending order was as follows: G2>G3>G4>G8>G7>G6>G1>G5.

Table 2. Mean (± standard deviation) microtensile bond strength of the 8 groups (n=10) in megapascals (MPa).

Group	1	2	3	4	5	6	7	8
Bond strength(Mean±SD)	0.51±0.18	6.57±1.15	2.96±1.02	1.89±0.94	0	1.18±0.79	1.29±0.89	1.34±0.77

SD: Standard deviation.

In the CGIC control group, all samples were broken during removal from the mold or placement in the universal testing machine. One-way ANOVA showed a significant difference in microtensile bond strength of the groups (P=0.01). Thus, pairwise comparisons were carried out using the Tukey’s test, which revealed that the bond strength in group 2 was

significantly higher than that in other groups (P≤0.001). The bond strength of group 1 (control) was significantly lower than that of RMGIC groups namely groups 2 (P≤0.001), 3 (P=0.00) and 4 (P≤0.001). Group 3 had significant differences with groups 1 (P≤0.001), 2 (P≤0.001), 6(P≤0.001), 7 (P≤0.001) and 8 (P≤0.001). Groups 6, 7 and 8 only had significant differences with groups 2 and 3. Although group 8 showed higher bond strength than groups 6 and 7, this difference was not significant. Maximum bond strength was noted in RMGIC bonded with ClearfilSE bond, which was significantly higher than the bond strength in other groups. Among self-cure GIC groups, maximum bond strength was noted in the use of universal adhesive in total-etch mode but the difference between this group and other CGIC groups was not significant (P>0.05).

Table 3 shows the frequency of different modes of failure. Of 60 samples that were subjected to scanning electron microscopic analysis (excluding the 20 lost specimens), 37 showed cohesive failure in GIC, and 23 showed mixed failure. No fracture in composite occurred in any sample.

Table 3. Frequency of different modes of failure based on scanning electron microscopic evaluation.

Lost Specimens	Frequency of Modes of Failure			Group
	Cohesive in Glass Ionomer	Cohesive in Composite	Mixed	
1	6	0	2	1
0	4	0	6	2
1	5	0	4	3
1	6	0	3	4
1	6	0	3	6
2	6	0	2	7
2	4	0	3	8

4. DISCUSSION

The bond strength of GIC to composite resin depends on the type of adhesive system (viscosity against surface wettability) [18, 19] and the type of glass ionomer [19]. This was also confirmed in our study. The maximum bond strength was achieved when RMGIC was used with a two-step self-etch bonding system, which was in agreement with the results of previous studies [5, 6, 12, 20].

The minimum bond strength was noted in the control groups. These findings may be due to the high viscosity of the composite resin, which prevents its optimal flow on the surface of the GIC sample without a wetting agent [21].

Difference in the type of GICs, conduction of thermocycling and water storage, and dimensions of composite and GIC samples may explain the differences between the results of this study and the previously reported literature [1, 4, 21 - 24].

Ansari et al. [23] found no significant difference in bond strength between RMGIC and CGIC following the application of self-etch adhesive. Difference between the results of Ansari et al. [23] and our findings may be due to the fact that they did not perform thermocycling. Anastasiadis et al. [22] showed

that water storage decreased the bond strength, which is due to the hydrolysis of the interface of GIC and composite resin following immersion in water. Based on this explanation, decreased bond strength after thermocycling in this study may be due to two factors namely hydrolytic degradation, and the difference in the coefficients of thermal expansion of GIC and composite resin.

It seems that the properties of the bonding agent can also affect the bond strength of GICs to composite resin. Type of monomer, mode of application (self-etch or total-etch), type of solvent, and viscosity of the bonding agent can all affect the bond strength of GICs to composite resins.

Universal adhesives are in fact self-etch single-step adhesive systems that can be used in self-etch or total-etch mode [25]. The bond strength of GICs to composite resin using universal adhesives has been previously evaluated in some studies [2, 4, 12]. Deepa *et al.* [4] used Single Bond Universal (3M, ESPE), which contains bis-GMA, HEMA, MDP, and acrylic/itaconic acid copolymer in water-alcohol solvent with a pH of 2.7 while G-Premio Bond contains MDP, 4-MET and MEPS monomers in acetone and water with a pH of 1.5. The presence of polyalkenoic acid in Single Bond Universal may be responsible for the stronger and more durable bond of dentin to the adhesive system [25].

The RMGIC bonded with a two-step self-etch adhesive system yielded significantly higher bond strength than other groups in this study. This difference might be related to the type of solvent, presence of HEMA, acidity of the bonding agent, or time of application of bonding agent.

The SE bond contains water solvent, which enhances surface wetting and results in a better flow of the resin on the surface. On the other hand, unpolymerized HEMA on the surface of RMGIC may enhance wetting and yield higher bond strength [26]. These two components (water and HEMA) may have a synergistic effect. Similarly, Kandaswamy *et al.* [27] showed that a mild self-etch adhesive (pH=2) yielded a stronger bond between GIC and composite resin compared with intermediate (pH=1.4) and strong (pH=1) adhesive systems. Application of a weak acid on the surface of GIC results in the availability of higher amounts of Na⁺, Ca²⁺ and Al³⁺ ions for bonding, which enable the formation of ionic bonds [23]. This could be responsible for higher bond strength achieved in this study.

Shorter application time of bonding agent (10 s versus 20 s) and the presence of acetone solvent with higher vapor pressure [23] may result in poor adaptation of resin to the surface, and decrease the penetration depth of G-Premio into the surface. One may assume that the presence of silane in G-Premio may increase its wettability. However, our study showed that the simultaneous presence of silane, other monomers, and acetone did not result in better wettability.

In etch and rinse application of G-Premio, our results showed no significant difference in bond strength of the two types of GICs to composite resin, which was in line with the results of Munari *et al.* [12]. According to Moghadam *et al.* [28] application of strong acid on the RMGIC surface results in the availability of lower amounts of cationic ions for a strong

bond between RMGIC and composite resin. Moreover, strong acids dissolve the surface of GIC and decrease its cohesive strength, resulting in a weaker bond. Kerby and Knobloch [29] showed that the application of acid on the RMGIC surface removed the air-inhibited layer, and adversely affected the chemical bonding. However, we found contrary results since the bonding agent used in this study contained 10-MDP and other acidic monomers that simultaneously etch the surface.

Evidence shows that mainly the failure mode of GICs is often cohesive rather than adhesive in bond strength tests [30]. The weak tensile strength of GICs may be responsible for this finding. The bond strength also depends on the presence of defects in the specimens [12], and void-free adaptation of composite to GIC [18]. As mentioned earlier, the authors designed a modified microtensile bond strength test to produce samples with fewer cracks and defects in the process of preparation. It seems that the difficult process of application of materials and removal of samples from two plexiglass molds measuring 12 x 1 x 1 mm and 6 x 1 x 1 mm might have created stress at the adhesive interface, leading to improper adaptation of composite resin to GIC, and creating some concerns with regard to the correct application of materials. A higher frequency of cohesive failure in the samples compared with mixed failure may further support this possibility. Thus, the bond strength values reported in such experimental studies are not a true representative of the adhesive bond strength, and rather indicate the tensile strength of GICs. Also, some studies discussed that cohesive failure was correlated with high bond strength [2,11].

Conduction of thermocycling was a strength of this study since it helped in better simulation of the clinical oral environment [31]. A total of 3000 thermal cycles were applied in this study, corresponding to 3 months of clinical service, which might be too short for accurate simulation of the clinical service of restorations. Moreover, thermal alterations induced by thermocycling cannot perfectly simulate the thermal alterations that occur in the oral cavity [32]; thus, a generalization of the results to the clinical setting should be done with caution.

Future studies are required to assess the effects of different generations of dentin bonding agents on bond strength of GICs to different types of composite resins, and primary and permanent enamel and dentin. Also, the effects of water storage and different solvents on the clinical service of different adhesives should be investigated in future studies. Furthermore, clinical studies are required to compare the efficacy of different bonding agents in the sandwich technique.

CONCLUSION

Within the limitations of this study, the results showed that the application of bonding agents significantly enhanced the bond strength of GICs to composite resin. The maximum microtensile bond strength was achieved when RMGIC was used with a mild two-step self-etch adhesive. Acid etching prior to the application of G-Premio universal adhesive is not necessary for the bonding of CGICs or RMGICs to composite resin.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Not applicable.

HUMAN AND ANIMAL RIGHTS

Not applicable.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

The data that support the findings of this research are available from the corresponding author [E.A] upon request.

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CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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