Research article

Microtensile bond strength of glass ionomer cements to a resin composite using universal bonding agents with and without acid etching

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ABSTRACT

Objectives: To assess the effect of two universal bonding agents on the microtensile bond strength (µTBS) of encapsulated conventional glass ionomers (CGICs) and resin-modified glass ionomers (RMGICs) to a resin composite, with or without the use of 35% phosphoric acid.
Methods: Four materials were used in this study: Riva Self-cure and Riva Light-cure; SDI and Equia Forte Fil and Fuji II LC; GC. The specimens were prepared in Teflon moulds with half the specimens for each GIC etched using 35% phosphoric acid (Ultra-Etch) and the remainder not etched. Each group was randomly subdivided into three groups, where the first two groups received an air-thin layer of bonding agent (G-Premio Bond = GPB or Clearfil Universal Bond = CUB) then light cured; and the third group had no bonding agent. For all groups, a nanohybrid composite (GC Kalore; GC) was placed incrementally on the GIC. Following 24 h immersion in distilled water, each block was embedded in epoxy resin in a cubic mould and sectioned by a cutting device. The stick specimens were then subjected to µTBS testing.
Results: The application of both universal bonding agents significantly enhanced the µTBS of all GICs (p < 0.001). Both RMGICs exhibited higher µTBS compared to that of CGICs (p < 0.001). The application of universal bonding agents with acid etching significantly increased the µTBS of both CGICs and RMGICs to resin composite in contrast to without acid etching.
Conclusion: Using 35% phosphoric acid for 15 s prior to the application of universal bonding agents improved the µTBS of GIC to resin composite.
Clinical significance: Using Universal bonding agents with 15 s acid etching may increase the bond strength of both CGICs and RMGICs to resin composite when utilising the sandwich technique.

1. Introduction

With the increased use of direct restorative materials by dental practitioners, newly developed resin composites are widely considered appropriate for anterior and posterior restorations [1, 2]. Among direct aesthetic restorative materials, resin composites are more durable [3], less degradable [4] and more resistant to fracture [5]. In order to compensate for polymerization shrinkage, using glass ionomer cement as a base is recommended when restoring a deep and large cavity [6]. Along with mechanical and thermal stresses that create contraction, internal stresses may lead to deformation and possible fracture of either the composite restoration or tooth structure [7].

Glass ionomer cements (GICs) are popular direct aesthetic restorative materials used in non-stress bearing regions [8], due to long-term fluoride release [9] and physiochemical adherence to tooth structure [10]. Furthermore, GICs are biocompatible with oral tissues [11, 12], have excellent bioactivity [3, 13], low cytotoxicity [7] and a similar coefficient of thermal expansion to that of tooth structure (dentin) [14]. To minimize polymerization shrinkage in large restorations, a substantial part of dentin is recommended to be replaced with GIC prior to the placement of resin composite. This is called the sandwich technique, which can also be referred to as the laminate or bilayered technique [6, 15]. This technique combines the advantages of both GIC and resin composite in a single restoration, decreases C-factor and polymerization shrinkage and increases clinical durability [15, 16].

There are two main types of Glass ionomer cements, conventional (CGICs) and resin-modified (RMGICs) [17]. Although these two types of GIC have variations in adhesion mechanism, setting reaction and sensitivity to moisture, both can be used as a base in the sandwich technique [17]. The drawbacks of CGICs, including early moisture sensitivity [18], low working time and slow setting reaction [8], have led to the development of RMGICs [7]. RMGICs have higher tensile and fracture strengths [7, 19], enhanced working time, rapid setting with light curing [20] and improved chemical solubility in comparison with CGICs [8]. Additionally, RMGICs have significantly improved cohesive strength and bond strength to tooth structure [7, 21]. However, the use of RMGICs in aesthetic and high stress-bearing areas is limited [7].

To achieve the best outcome when employing the sandwich technique, the bond strength of GIC to resin composite should be strong. Some studies [22, 23] have demonstrated that acid etching enhances the bond strength of CGICs to resin composites. Others [24, 25], reported no significant difference in bond strength of resin composite to etched or non-etched GICs. Pamir *et al.* [26] determined the effect of different surface treatment modalities on the bond strength of resin composites to GICs. It was shown that RMGICs have significantly higher bond strength to resin composites compared to that of CGICs, which can be further enhanced with 30 s acid etching prior to bonding application [26].

Dental bonding agents are developed to promote durability of restorations and to reduce the number of procedural steps making them more user friendly [27]. Studies have demonstrated that applying bonding agents results in improvement of the bond strength of GIC to resin composite [28, 29]. Dental bonding agents have evolved from non-etch to total-etch (4th and 5th generation), then self-etch (6th, 7th and 8th generation) systems [30]. The most recent bonding agents are the universal bonding agents (also known as multi-mode or multi-purpose adhesives), as they can be used in both total-etch (TE) and self-etch (SE) modes on dentin or enamel [30]. However, due to the limited number of studies conducted on this topic [24, 25], it is unclear which mode of universal bonding agents enhances the microtensile bond strength (µTBS) of GIC to a resin composite. Recently, Munari *et al.* [25] have shown that GIC etching promoted higher µTBS to resin composite with a universal bonding agent (Single Bond Universal, 3M-ESPE, USA) than with a TE bonding agent (Adper Single Bond 2, 3M-ESPE, USA). They also demonstrated that acid etching of GICs is not necessary to enhance the µTBS of the universal bonding agent, and negatively affects the µTBS of the TE bonding agent. In a recent study Kermanshah *et al.* [24] demonstrated that µTBS of RMGIC to resin composite using a SE bonding agent (Clearfil SE Bond, Kuraray, USA) was significantly higher than that of using a universal bonding agent (G-Premio Bond, GC, Japan) in both SE and TE modes. Additionally, they showed that acid etching prior to the application of G-Premio bonding agent is not necessary to promote µTBS of CGICs or RMGICs to resin composite [24].

There are number of published studies [24, 25] investigating the efficacy of acid etch and universal bonding agents on the µTBS of CGICs and RMGIC to resin composite, however there is no consensus on the type of surface treatment modalities on CGICs and RMGICs. Therefore, this study aimed to determine the effect of universal bonding agents [G-Premio Bond (GPB) or Clearfil Universal Bond (CUB)] on the µTBS of four encapsulated CGIC and RMGIC to resin composites, with or without using 35% phosphoric acid. The null hypothesis was that neither type of universal bonding agents nor the application of acid etch have any effect on the µTBS of the CGICs and RMGICs to a resin composite.

2. Materials and methods

Four GICs, one resin composite and two bonding agents were used in this study (Table 1). The µTBS test was performed based on the recommendation of Armstrong *et al.* [31] with some modification on preparing the specimens. The experimental regimen is shown in Figure 1. Overall, 24 cubic specimens were prepared using a Teflon mould (5 mm width, 6 mm height and 10 mm length) containing a

Table 1. Materials description.

Materials	Manufacturer	LOT number	Type	Composition
Equia Forte Fil	GC, Tokyo, Japan	1604041	CGIC	Fluoro-alumino-silicate glass/Polybasic carboxylic acid/ Polyacrylic acid/ Distilled water
Riva Self-cure	SDI, Victoria, Australia	1081615F	CGIC	Fluoro-aluminosilicate glass/Polyacrylic acid/ Tartaric acid
Fuji II LC	GC, Tokyo, Japan	1607141	RMGIC	Aluminium-fluoro-silicate glass/Poly-HEMA
Riva Light-cure	SDI, Victoria, Australia	1082160EG	RMGIC	Fluoro-aluminosilicate glass/Polyacrylic acid
Ultra-Etch	Ultradent, Utah, USA	BGTW2	Acid Etch	35% Phosphoric acid
Clearfil Universal Bond	Kuraray, Tokyo, Japan	900035	Universal Bonding Agent	Bis-GMA, HEMA, MDP, hydrophilic aliphatic dimethacrylate, ethanol, water, initiator, silica, silane coupling agent
G-Premio Bond	GC, Tokyo, Japan	1802103	Universal Bonding Agent	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, initiator, silica
GC Kalore	GC, Tokyo, Japan	1601201	Universal Resin Composite	UDMA/Dimethacrylate urethane DX-511/Bis-EMA/Fluoro-aluminium-silicate glass/Prepolymerized filler/Silicon dioxide

4-MET, 4-methacryloyloxyethyl trimellitate; Bis-GMA, bisphenyl A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxy-decyl dihydrogen phosphate; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; UDMA, Dimethacrylate urethane; Bis-EMA, Ethoxylated dimethacrylates.

compartment for GIC and resin composite (5 mm width, 6 mm height and 5 mm length) insertion.

The encapsulated GICs were activated according to the manufacturer's instructions for 10 s. Riva-self cure and Riva-light cure (SDI, Victoria, Australia) were activated on the Amalgamator (Ultramat 2, SDI Limited, Bayswater, Australia), whereas, Equia Forte Fil and Fuji II LC (GC, Tokyo, Japan) were activated on the Dental Capsule Mixer (CAPSULE MIXER CM-II™, Taipei, Taiwan). The GICs were then inserted into the mould, and covered with a Mylar strip. In the case of light-cure GICs, specimens were light-cured for 20 s with 1500 mW/cm^2 (Radii plus, SDI, Victoria, Australia). Half the specimens for each GIC group were left non-etched and the other half etched using 35% phosphoric acid (Ultra-Etch, Ultradent, USA) for 15 s followed by 15 s of rinsing and drying. Each group was randomly subdivided into three groups. The first two groups received a thin layer of bonding agent (GPB or CUB) and were light cured for 20 s, whereas, the third group received no bonding agent. For all groups a universal nanohybrid composite (GC Kalore) was placed incrementally (1.5–2 mm layers) on the GICs and light cured for 20 s. Finally, a cubic block (10 × 5 × 5 mm^3) in each of the 24 groups was customized. The blocks were removed from the moulds and stored in an incubator with 100% humidity in 37 °C for 24 h to allow for polymerization maturation. After 24 h immersion in distilled water, each block was embedded into epoxy resin in a cubic mould (3 cm width, 3 cm length and 1.5 cm height). The blocks were later sectioned by a cutting device (Mecatome T180, Presi, France) five times horizontally, then four times vertically in order to make as many stick specimens as possible (1 mm width, 1 mm height and 10 mm length), this is illustrated in Figure 2. Ten intact stick specimens in each of the 24 groups were chosen to be tested. The prepared groups are shown in Table 2.
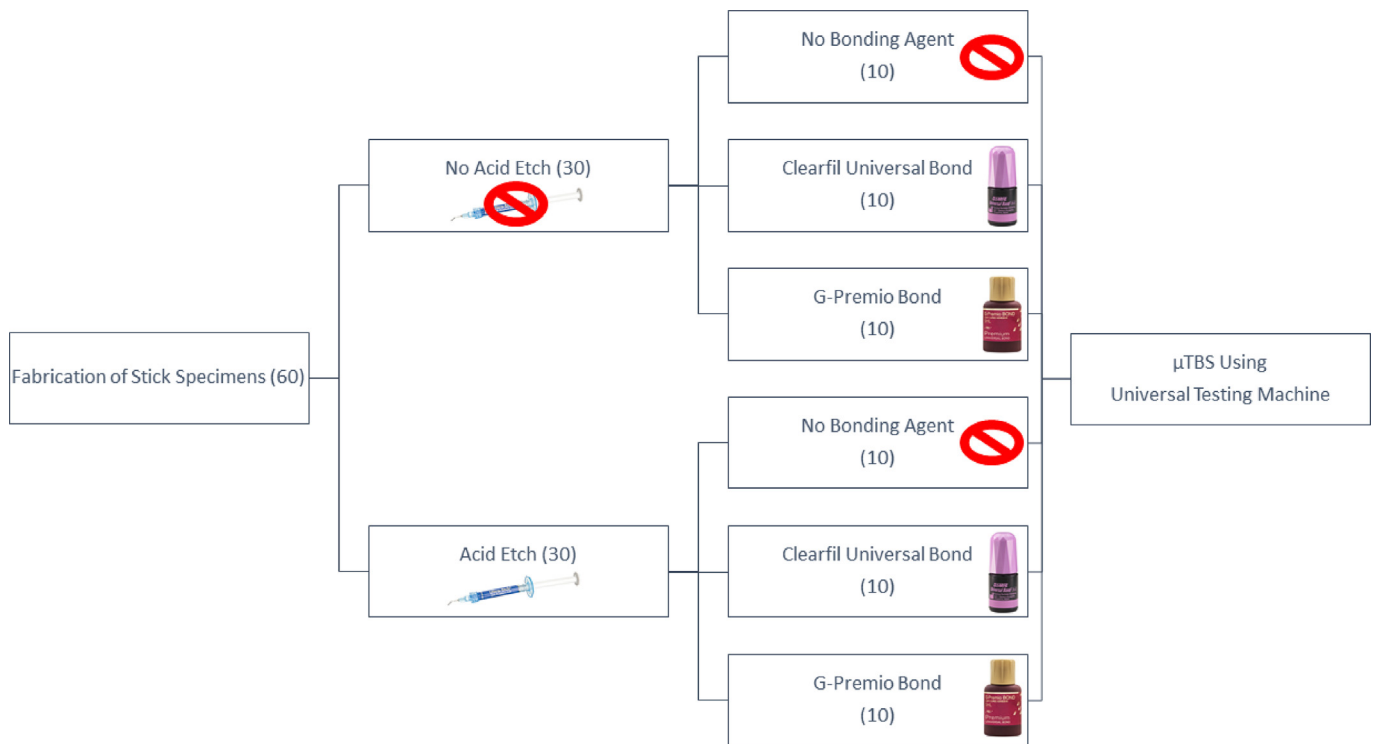
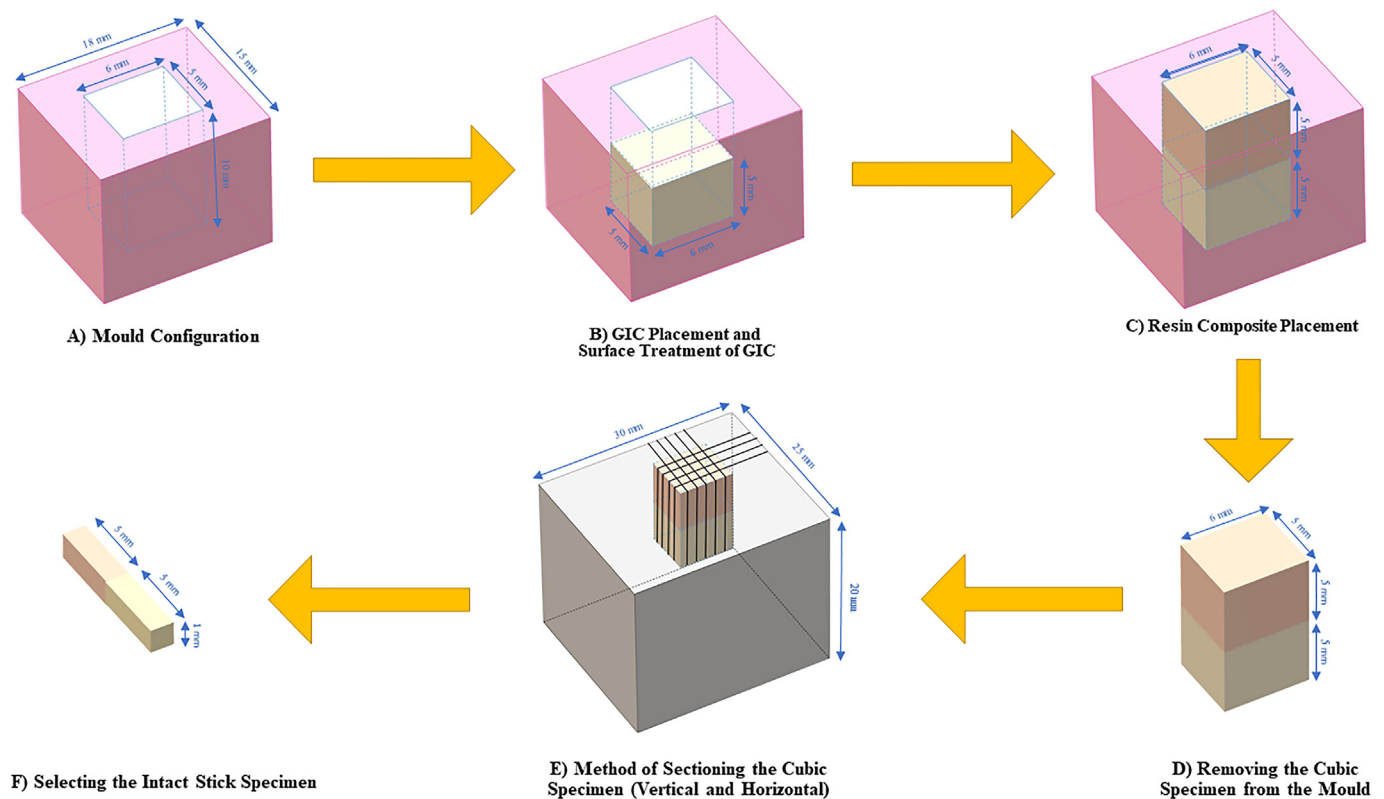**Figure 1.** Treatments and microtensile measurement regimen.

Figure 2. Schematic drawing of the specimen preparation.

2.1. Microtensile bond strength test (μTBS)

Each stick specimen was fixed into a microtensile jig using a cyano-acrylate adhesive, attached to a universal testing machine and subjected to the microtensile bond strength with a crosshead speed of 0.5 mm/min.

The maximum force to fracture was recorded in N, and μTBS was calculated using Eq. (1):

$$\mu\text{TBS (MPa)} = \text{Force (N)} / \text{SA (mm}^2) \qquad \text{(Equation 1)}$$

Table 2. Mean ± SD (MPa) of µTBS of all GICs to resin composite using two universal bonding agents with and without acid etching.

Materials	Without Etching		
	Control (No Etch or Bond)	Clearfil Universal Bond	G-Premio Bond
Equia Forte Fil	1.48 ± 0.57[Aa]	3.33 ± 0.66[Ab]	3.83 ± 0.93[Ab]
Riva Self-cure	1.99 ± 0.21[ACa]	5.77 ± 0.80[Bb]	3.85 ± 0.62[Ac]
Fuji II LC	3.73 ± 0.43[Ba]	6.61 ± 0.77[BCb]	9.23 ± 0.64[Bc]
Riva Light-cure	3.21 ± 1.14[BCa]	7.25 ± 0.83[Cb]	6.34 ± 0.79[Cb]
Materials	**With Etching**		
	Control (Only Etch)	**Etch + Clearfil Universal Bond**	**Etch + G-Premio Bond**
Equia Forte Fil	2.52 ± 0.72[Aa]	5.36 ± 0.91[Ab]	6.32 ± 0.70[Ab]
Riva Self-cure	2.95 ± 0.44[Aa]	7.44 ± 0.70[Bb]	6.88 ± 0.64[Ab]
Fuji II LC	5.40 ± 0.61[Ba]	11.39 ± 0.96[Cb]	11.98 ± 1.48[Bb]
Riva Light-cure	4.96 ± 0.59[Ba]	10.47 ± 0.63[Cb]	10.17 ± 0.69[Cb]

Different upper-case letters show significant difference between µTBS of all GIC to resin composite in each column.
Different lower-case letters show significant difference between µTBS of each GIC to resin composite in each row.

The cross-sectional adhesion area (SA) was measured using a digital caliper (Mitutoyo, Kawasaki, Japan) with 0.01 mm accuracy.

2.2. Statistical analysis

The data were analyzed using SPSS, version 21 (SPSS Incorporation, Chicago, IL, USA). Kolmogorov Smirnov test was employed for assessing normality assumption of data and parametric analysis for comparison. Independent t-test was used for pair comparison of related subgroups with and without etching when considering the type of GIC. One-way ANOVA and Tukey's test was used for comparing subgroups with and without etching. Two-way ANOVA was applied to determine interactions between the groups with and without etching and their subgroups. The level of significance was set at 0.05.

3. Results

The mean µTBS (MPa) and standard deviation for all groups is shown in Tables 2 and 3. The maximum µTBS value was recorded for Fuji II LC when used with G-Premio bond in the etching mode (11.98 ± 1.48); whereas, the minimum µTBS recorded belonged to Equia Forte Fil used without any surface treatment (1.48 ± 0.57). Table 3 demonstrates that the application of universal bonding agents using acid etch significantly increased the µTBS of both CGICs and RMGICs to resin composite, in comparison to that of the non-etching mode. Additionally, using acid etch alone significantly increased the µTBS of only RMGICs to resin composite.

Table 2 demonstrates that the application of universal bonding agents significantly promoted the µTBS of CGICs and RMGICs in comparison to their control groups (p < 0.001). The µTBS of RMGICs to resin composites using universal bonding agents was higher than that of CGICs (p < 0.001), except for Riva Self-cure and Fuji II LC using CUB without acid

etching. Additionally, regardless of applying any bonding agents, the µTBS of RMGICs to resin composites was higher than that of CGICs. It should be noted that the application of GPB on Fuji II LC resulted in significantly higher µTBS than that of other GICs (Equia Forte Fil, Riva Self-cure, and Riva Light-cure) to resin composite. Conversely, the application of CUB on Riva Self-cure resulted in significantly higher µTBS than the other CGIC (Equia Forte Fil) to resin composite.

4. Discussion

Resin composite polymerisation induces stress at the interface of the restoration and cavity walls. This stress may result in debonding of the restoration from the cavity walls or tooth structure [32]. To reduce adverse effects from shrinkage stress, the sandwich technique is used. In this technique, a considerable part of the lost tooth structure is replaced with GIC, covering most of the exposed dentin to form the cervical seal [33]. The pairwise comparison showed a significant difference in the µTBS between the tested universal bonding agents and acid etching procedures, revealing that use of acid etching and bonding increase the bond strength of GIC to resin composite. Furthermore, applying acid etch without any bonding agent on both RMGICs (Fuji II LC and Riva Light-cure) enhanced the bond strength to resin composite. In agreement with our results, Navimipour et al. [23] studied the effects of acid and laser etching on shear bond strength of CGIC and RMGIC to a resin composite, They reported that the application of 35% phosphoric acid gel (Scotchbond Etchant, 3M ESPE, USA) for 15 s on RMGIC (Fuji II LC, GC Corporation, Japan) lead to a significantly stronger bond between GIC and resin composite. The authors speculated that in the process of acid etching, phosphoric acid attacks the matrix of the GIC resulting in a rough and porous surface, providing a retentive surface to enhance adhesion to composite resin [23]. Pamir et al. [26] investigated the effects of acid etching and adhesive applications on the bond strength between resin composite, CGIC (Ketac™ Molar Quick Applicap™, 3M ESPE, Germany) and RMGIC (Photac™ Fil Quick Applicap™, 3M ESPE, Germany). The study demonstrated that etching GIC increased the bond strength of resin composite, where bond strength of RMGIC was higher than CGIC. Another study [24] evaluating the µTBS of RMGIC (Fuji II LC, GC Corporation, Japan) and CGIC (Fuji IX, GC Corporation, Japan) to a resin composite (Filtek Z250, 3M/ESPE) using universal bonding agents in different modes, dictated that the maximum bond strength was achieved when RMGIC was used with a two-step self-etch.

Conversely, other studies reported controversial results [22, 25] when assessing the effect of acid etching with 37% phosphoric acid (Acid Gel, Dentalville do Brasil LTDA, Brazil) for 10 s using various adhesive systems (Single Bond Universal and Adper Single Bond 2, 3M/ESPE, USA), on the µTBS of CGIC (Riva Self-cure, SDI, Australia) to a nano-filled resin composite [25]. They concluded that acid etching can dissolve the CGIC matrix and decrease the adhesive wetting surface [25]. Otsuka et al. [22] evaluated the effect of surface treatment by 35% phosphoric acid (Gel Etchant, Kerr, USA) of CGIC (Fuji IX GP, GC Corporation, Japan), and two RMGICs (Fuji II LC and Fuji Filling LC, GC Corporation, Japan) on the surface free energy and bond strength of resin composite. The results of this study showed that the surface treatment of CGIC promoted higher bond strength to a resin composite, whereas the surface treatment of RMGIC revealed a negative effect on the shear bond strength. This

Table 3. Mean ± SD (MPa) of µTBS of all GICs to resin composite using two universal bonding agents with and without acid etching (comparing different treatments using etchant and bonding agents).

Materials	Control (No Etch or Bond)	Only Etch	P Value	Clearfil Universal Bond	Etch + Clearfil Universal Bond	P Value	G-Premio Bond	Etch + G-Premio Bond	P Value
Equia Forte Fil	1.48 ± 0.57	2.52 ± 0.72	.958	3.33 ± 0.66	5.36 ± 0.91	.000	3.83 ± 0.93	6.32 ± 0.70	.000
Riva Self-cure	1.99 ± 0.21	2.95 ± 0.44	.983	5.77 ± 0.80	7.44 ± 0.70	.004	3.85 ± 0.62	6.88 ± 0.64	.000
Fuji II LC	3.73 ± 0.43	5.40 ± 0.61	.021	6.61 ± 0.77	11.39 ± 0.96	.000	9.23 ± 0.64	11.98 ± 1.48	.000
Riva Light-cure	3.21 ± 1.14	4.96 ± 0.59	.022	7.25 ± 0.83	10.47 ± 0.63	.000	6.34 ± 0.79	10.17 ± 0.69	.000

finding may be attributed to the removal of a resin-rich layer at the surface of the RMGIC [22].

Optimum bonding depends on the wettability of the GIC surface. In our study, the minimum µTBS belonged to the control groups followed by CGIC (Equia Forte Fil) without etching. In the results of this study, the application of etching agents without any bonding agents on CGICs does not enhance the µTBS. These outcomes may be due to the high viscosity of the resin composite, which prevents ideal flow on the surface of the CGIC without a wetting agent [34]. Sadeghi *et al.* [7] focused on the shear bond strength of a resin composite to RMGIC (Light-Cured Universal Restorative, GC Corporation, Japan) using three different resin-based adhesives (OptiBond Solo Plus (TE adhesive), OptiBond XTR (two-step SE adhesive), and OptiBond All-in-One (one-step SE adhesive), Kerr, Italy) and a glass ionomer-based adhesive (Fuji Bond LC, GC Corporation, Japan). They [7] concluded that adhesion between CGIC and resin composite is less than that of RMGIC due to the water sensitivity of CGIC and lack of chemical bonding between these materials. They highlighted the lack of significant differences in shear bond strength of resin composite to RMGIC utilizing different generations of resin-based and glass ionomer-based adhesives [7]. In the current study, the bond strength between the CGICs and resin composite were significantly lower than that of the RMGIC groups. The presence of resin components such as hydroxyethyl methacrylate (HEMA) in RMGIC, could explain the increase in bond strength between RMGIC and resin composite. HEMA is a low-weight hydrophilic monomer which wets the substrate and is highly soluble in water, ethanol and acetone, and it is easily incorporated into adhesive formations [35]. The HEMA component in RMGIC creates stronger chemical adhesion to the composite system using a resin-based bonding agent [7]. Furthermore, the application of the bonding agents enhances the wettability of the RMGIC, hence increasing its bond to the resin composite. The similar chemical components between the RMGIC and resin composite enables a strong bond [29].

Based on the results of our study, the application of universal bonding agents with or without etching significantly promoted the µTBS of CGICs and RMGICs to resin composite in comparison to the control groups. The composition of universal bonding agents is different from the current SE systems as they contain monomers that are capable of producing chemical and micromechanical bonds to substances [30]. Universal bonding agent is based on a combination of hydrophilic monomers including HEMA and hydrophobic monomers such as D3MA (decandiol dimethacrylite) and intermediate monomers such as Bis-GMA (Bisphenol A-glycidyl methacrylate) [30]. The hydrophilic ends interact with GIC while the hydrophobic ends interact with methylmetacrylate-based restorative materials i.e. resin composite [35]. Most universal bonding agents contain carboxylate and phosphate monomers which may influence bond strength. MDP (methacryloydecyl dihydrogen phosphate) is a hydrophilic monomer with mild etching properties enabling universal bonding agents to be used with any etching techniques. The highest µTBS of universal bonding agents to surfaces with previous etching may be the result of the chemical union of MDP and calcium ions from GIC [25]. The results of the µTBS test do not provide a reflection of the material itself, yet indicate the behavior of the bonding system in various configurations of the test. Changing the test materials can lead to different outcomes and as this is an in-vitro study, the results would not necessarily be the same as in clinical practice. Therefore, continuation in the form of randomized controlled clinical trials (RCTs) are required to formulate a clinically relevant conclusion. Future studies should evaluate the effects of different generations of bonding agents on bond strength of GICs to different types of resin composite.

5. Conclusion

Based on the results of the current study, we can conclude that the application of universal bonding agents with 15 s etching may improve the µTBS in comparison to no etching. When using etchant, µTBS of all RMGICs to resin composites were higher than that of CGICs. It seems that acid etching prior to the application of universal bonding agents is necessary to enhance the µTBS of GIC to resin composite. However, randomized controlled clinical studies are required to prove clinically relevant conclusions.

Declarations

Author contribution statement

Nima Farshidfar, Mahya Agharokh: performed the experiments; analyzed and interpreted the data; contributed reagents, materials, analysis tools or data; wrote the paper.

Maryam Ferooz, Rafat Bagheri: conceived and designed the experiments; analyzed and interpreted the data; contributed reagents, materials, analysis tools or data; wrote the paper.

Data availability statement

Data will be made available on request.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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