Original Article

Microshear Bond Strength of High-Viscosity Glass Ionomer Cement and Resin Cements: An *in vitro* Study

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ABSTRACT

Background: While the advent of self-adhesive resin cements has simplified indirect restoration luting by reducing technique-sensitivity, the clinical longevity of these restorations remains fundamentally dependent on the bond strength achieved by different resin cement types to the restoration material.

Purpose: The aim of this study is to investigate the microshear bond strength of three types of resin cements (RCs) to high-viscosity glass ionomer cements (HVGICs).

Materials and Method: In an *in vitro* study, sixty cylindrical specimens were prepared from two HVGICs (EQUIA Forte Fil and Riva). Then, a polyvinyl chloride microtube (with 0.7mm diameter and 0.5mm height) was placed on each sample surface and was filled using various types of RCs, including conventional RC (Dou-Link), self-etch RC (Panavia F2), and self-adhesive RC (TheraCem). The μSBS of the specimens was measured after 24 hrs. and analyzed with two-way ANOVA and Tukey test (p Value< 0.05).

Results: According to the result of the two-way ANOVA test, the RCs showed a significant effect on μSBS (p< 0.0001) while no significant effect was observed in HVGIC on the μSBS (p= 0.325). For both HVGICs, there was a significant difference between the μSBS of Panavia F2 and the other types of RCs (p< 0.0001) while no statistically significant difference was found between Dou-Link and TheraCem (p= 0.515). No significant difference was observed between the two HVGICs according to the Tukey test results (p= 0.325).

Conclusion: Self-adhesive resin cements showed higher bond strength than other resin cement. Moreover, different types of HVGICs make no difference in the RCs bond strength when used as the core-build-up.

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Introduction

In recent years, resin cement (RC) has been widely the material of choice for luting in indirect restorations [1]. It shows numerous advantageous properties for cementation such as favorable esthetics, excellent mechanical behavior, and strong bonding to the tooth structure [2-3]. There are different types of RCs used in restorative dentistry including conventional and self-adhesive RC [4]. Conventional RCs contain an adhesive system that can be applied in etch-and-rinse or self-etch modes, based on adhesion agent [4]. Self-adhesive RCs were developed to simplify the cementation process by combining adhesive and cement functions into a single step, eliminating the need for dental substrate pretreatment. This innovation reduces procedural complexity, minimizes the risk of operative errors, and has gained widespread acceptance among clinicians [4-5]. This has increased their popularity in restorative dentistry by decreasing technique sensitivity and chair side time [1]. Despite the advantages of RCs, the bond strength of different types of RCs to restoration materials has been always a key factor for the clinical success of indirect restorations [1].

McLean and Wilson were the first who introduced

glass ionomer cement (GIC) to the dentistry in the 1970s [5]. GICs are based on the reaction between a powder, calcium fluoro aluminosilicate (FAS) glass particles, and a liquid that can be an aqueous polyacrylic acid or a copolymer of polyacrylic and maleic or itaconic acid [6-7]. GIC shows bioactivity properties and forms chemical bonds to enamel and dentine by ionic bonding mechanisms [8]. GICs are widely used in various dental applications due to their advantageous properties including low toxicity, biocompatibility, and high adhesion to hard dental tissues, decreased shrinkage, and fluoride release [6, 9]. According to extensive longitudinal studies and laboratory research, C-GICs were considered unsuitable for broad application as permanent restorations due to their inadequate mechanical properties. They were found to be less durable and more prone to failure compared to resin composite, particularly when used for occlusal or approximal restorations in posterior teeth [10].

Recently, a new generation of GIC, known as high-viscosity glass ionomer cement (HVGIC) has been developed [11]. While retaining the high biocompatibility of conventional GICs, HVGIC exhibits superior mechanical properties- including enhanced resistance to bending, compression, and abrasion [12] as well as greater mechanical strength, improved marginal sealing, and longer clinical durability, making it a preferred alternative to traditional GIC [13]. The advancement of HVGIC technology led to the development of EQUIA Forte, a glass-hybrid system that combines larger glass-filler particles with smaller, highly reactive fillers. This formulation improves compressibility, reduces stickiness for easier handling, and increases flexural strength [14]. EQUIA Forte Fil is a high-viscosity material featuring micron-sized FAS fillers with high reactivity and molecular weight. These fillers release elevated levels of metal ions, reinforcing the polyacrylic acid matrix via cross-linking, thereby enhancing mechanical properties and fluoride release [15-16].

Joshi *et al.* [16] compared the mechanical properties of various GIC-based restorative materials and found that EQUIA Forte demonstrated the highest compressive and flexural strength. These advancements have expanded its applications to high-stress scenarios such as posterior load-bearing restorations and core build-ups [12, 17]. Clinical studies have reported successful outcomes with HVGIC in premolar load-bearing cavities [18], Class I/II restorations [19-20], hypomineralized teeth [21], and non-carious cervical lesions [21]. However, no research has yet investigated its bond strength as a core build-up material when used with RCs as luting agents.

The goal of this study was to investigate the microshear bond strength of different types of RC to HVGICs.

The null hypotheses of this study were that the type of HVGICs would not affect the microshear bond strength of the RC, and that different types of resin composites would have similar bond strength.

Materials and Method

Three types of RCs were investigated including Duo-Link (Bisco, Schaumburg, IL, US) as a conventional RC, Panavia F2.0 (Kuraray Noritake Dental, NY, NY, US) as a self-etch RC, and TheraCem (Bisco, Schaumburg, IL, US) as a self-adhesive cement. Moreover, two different HVGICs were studied including EQUIA Forte Fil (GC, Tokyo, Japan) and Riva (SDI, Bayswater, Australia). Table 1 reports the details of materials, abbreviations, manufacturers, and compositions.

Sample preparation

Following approval of the study design by the Research Ethics Committee of Shiraz University of Medical Sciences (Protocol #IR.SUMS.DENTAL.REC.1399.056), 60 cylindrical specimens of HVGIC were prepared in a stainless-steel mold with a diameter of 4mm and a height of 6mm. Each HVGIC brand was used in preparing thirty specimens and thus, two groups were formed.

In Group 1 (1a, 1b, 1c n= 30); each sample was prepared in a capsule containing EQUIA Forte Fil that was mixed mechanically with a trituration rate of 4200 for 10 s according to the manufacture's recommendations. Then, its special applier was used to inject EQUIA Forte Fil into the standardized cylinder on top of a glass slab. On top of the EQUIA Forte Fil, a Mylar matrix band was placed and covered with a glass slab to make sure that the specimen surface was smooth and parallel to the opposite surface of the mold and then, the mold was turned over. After making sure that the specimens were set, the mold was removed and EQUIA Forte Coat was applied to the specimens and then, they were light-cured for 10 seconds. A polyvinyl chloride microtube (with

Table 1: The brand names, manufacture, chemical composition of the materials used in this study

Brand/ Manufacturer	Composition	Manufacture
Panavia F2.0	A paste: Bis-GMA, TEGDMA, dimethacrylate, 10-MDP, silanized Ba glass filler, silanized colloidal silica, photo-initiator, chemical initiator B paste: Bis-GMA, dimethacrylate, silanized Ba glass filler, silanized colloidal silica, silanized NaF, chemical accelerator, pigment. ED Primer II: Primer A: HEMA, MDP,5-NMSA, water, accelerator. Primer B: 5-NMSA, accelerator, water, sodium benzene sulphinate	Kuraray Medical, Okayama, Japan
Duo-Link	Base:bis-GMA, triethyleneglycoldimethacrylate urethane dimethacrylate, glass filler Catalyst: bis-GMA, triethyleneglycoldimethacrylate, glass filler	Bisco, Schaumburg, U.S.A.
TheraCem	Calcium base filler, silanated nonreactive fillers, methacrylate monomers containing phosphoric acid groups, methacrylate monomers, ytterbium fluoride, initiators	Bisco, Schaumburg, U.S.A.
Riva self-cure	Fluroaluminosilicate glass, 2-hydroxylethyl methacrylate, polyacrylic acid, 2, 2, 4 trimethyl hexamethylene dicarbonate, proprietary ingredient	SDI, Victoria, Australia.
EQUIA Forte Fil	Powder: Fluroaluminosilicate glass, polyacrylic acid, iron oxide Liquid: polybasic carboxylic acid, water	GC, Tokyo, Japan.
EQUIA Coat	Methyl methacrylate, multifunctional methacrylate, camphor quinone	GC, Tokyo, Japan
Phosphoric acid etching	A 37% phosphoric acid gel etchant	Denfil/Vericom, Ltd., Korea
Adper Single Bond 2	HEMA, water, ethanol, Bis-GMA, dimethacrylates, amines, methacrylate functional copolymer polyacrylic and polyitaconic acids, nanometer-diameter spherical silica particles	C 3M ESPE, St Paul, MN, USA

the diameter of 0.7mm and the height of 0.5mm) was placed on each sample surface. Afterward, three different subgroups (n= 0) were formed, according to the type of RC used including Group 1a (n= 10): EQUIA Forte Fil + Dou-Link RC, Group 1b (n= 10): EQUIA Forte Fil + Panavia F2 RC, and Group 1c (n= 10): EQUIA Forte Fil + TheraCem RC.

In the groups in which Dou-Link has been applied and before cement placement, 37% phosphoric acid was used to etch the surface for 15 seconds and then, the surface was rinsed with water for 15 seconds and thoroughly air-dried. Two coats of Adper Single Bond 2 were applied to the HVGIC surface and air-dried for 5 seconds such that the solvent was thoroughly evaporated and finally, light-cured for 20 seconds. In the groups in which TheraCem has been used, the mixed cement was directly bonded to the HVGIC surface. In the groups in which Panavia F2 has been applied, ED primer II A and B were mixed in an equal ratio and then, applied to the HVGIC. Afterwards, it was left in place for 30 s and gently air dried. Paste A and B were mixed in an equal ratio and then, applied the cement on the surface of GI and light cured for 20s.

In Group 2 (2a, 2b, 2c n= 30); for each sample, a capsule of Riva was prepared and inserted into the cylinder similar to the procedure of the EQUIA Forte Fil. Three different subgroups (n= 10) were formed including Group 2a (n= 10): Riva + Dou-Link RC, Group 2b (n= 10): Riva + Panavia F2 RC, and Group 2c (n= 10): Riva + TheraCem RC.

The RCs were applied similar to the of the EQUIA Forte Fil group. For all curing steps, a Bisco Dental VIP Junior light-curing unit was employed, maintaining a light intensity of 600mW/cm². The intensity was verified using a radiometer after every fifth curing procedure.

Microshear Bond Strength Testing

After preparation, each sample was kept in distilled water storage containers at room temperature for 24 hr. before microshear bond strength (μSBS) testing. The tubes around the RC cylinders were gently cut using a surgical blade and removed completely. Data collection bias was avoided by blinding during testing specimens. Afterward, the μSBS was evaluated using a universal testing machine (Instron Z020; Zwick Roell, Ulm, Germany) while a wire loop was positioned at the interface of dentine/ cement at a 1 mm/min constant speed. The μSBS was measured in Newtons (N) and recorded in megapascals (MPa). One operator performed all measurements at the same time using the same device.

Failure Mode Analysis

After the bond strength measurement, the debonded specimens were observed under a stereomicroscope (Zeiss OPM1; Carl Zeiss, Oberkochen, Germany) at 40× magnification, and failures were classified as adhesive failure (when there was a failure between the HVGIC and RC), cohesive failure (when there is a failure in the

HVGIC or RC), and mixed adhesive (when both adhesive and cohesive failures occur) **Error! Reference source not found.**.

Statistical analysis

The data was analyzed with the Kolmogorov-Smirnov test to confirm its normal distribution. Using a two-way analysis of variance (ANOVA) model, the effects of the type of the HVGIC and the type of RC were evaluated. Post-hoc Tukey HSD test was used for subgroups analysis. Data were analyzed using the SPSS software package (SPSS, ver. 17.0; SPSS, Chicago, IL, USA). If the p-value was 0.05 or lower, the result was considered statistically significant.

Results

Table 2 shows the mean and standard deviation values of the experimental groups. According to the result of the two-way ANOVA test, the RCs showed a significant effect on µSBS ($p< 0.0001$) while no significant effect was observed in HVGIC on the µSBS ($p= 0.325$). Moreover, the interactions between the RC and HVGIC showed no significant effect on the µSBS ($p= 0.739$).

For both HVGICs, Panavia F2 revealed the highest value of µSBS, while TheraCem showed the lowest µSBS value. Post-hoc Tukey test also revealed a significant difference between the mean values of Panavia F2 and the other types of RCs ($p< 0.0001$), while the statistical analysis showed no significant difference between Dou-Link and TheraCem ($p= 515$). Furthermore, the Tukey test showed that there was no significant difference between the two HVGICs ($p= 325$).

Failure mode analysis

Failure mode analysis is presented in Table 3. In the group in which Panavia F2 has been applied, low adhesive failure was observed and most failures were cohesive which is consistent with the higher µSBS values obtained. The results of the groups in which Dou-Link and TheraCem has been used, showed the highest number of adhesive failures, suggesting the low quality of the bonding interface.

Table 2: Mean±standard deviation of microshear bond strength values (MPa) for different groups

	Duo-Link	TheraCem	Panavia F2
EQUIA Forte Fil	8.09±1.58 A,a	7.55±1.35 A,a	12.39±1.13 A,b
Riva	8.67±1.30 A,a	8.17±1.14 A,a	12.35±2.18 A,b

Different uppercase letters in columns and lowercase letters in rows indicate statistically significant difference ($p< 0.05$)

Discussion

HVGIC contains anhydrous polyacrylic acid of high molecular weight, fiberglass particles and a high powder-to-liquid mixing ratio which improves its physiomechanical properties such as compressive strength, wear resistance, and easier application [22]. Moreover, they show optimal biocompatibility and no polymerization shrinkage [23]. These properties make HVGIC an interesting choice for the core build-up materials in anterior and posterior teeth [24-25]. However, to increase the bond strength of ceramic material to the core materials, an appropriate cement is required. Since RC is a popular cementation material, it is necessary to conduct studies that involve bonding strength between HVGICs and different RCs.

In this *in vitro* study, we evaluated the µSBS of three RCs (Dou-Link, Panavia F2, and TheraCem) to two HVGICs (Riva and EQUIA Forte Fil). Riva and EQUIA Forte Fil were selected because they differ in the chemical composition of their fillers; i.e. 'ionglass' fillers reinforce Riva while ultrafine glass particles is applied to reinforced EQUIA Forte Fil [21, 26]. According to the obtained results, the first null hypothesis was accepted, that is the type of HVGICs does not influence the bond strength of RCs to HVGIC. Hence, it seems that the type of filler in HVGICs has no effect on their bond strength to RCs.

The second null hypothesis is rejected; i.e. the bond strengths of HVGIC to the RCs are independent of the type of cement. The present study investigated three RCs including Dou-Link (conventional RC), Panavia F2 (self-etching RC), and TheraCem (self-adhesive RC).

Table 3: Failure mode (%) of the groups, after the microshear test

Failure mode analysis	Group 1			Group 2		
	1a: EQUIA Forte Fil+Dou-Link	1b: EQUIA Forte Fil+Panavia F2	1c: EQUIA Forte Fil + TheraCem	2a: Riva+ Dou-Link	2b: Riva+ Panavia F2	2c: Riva+ TheraCem
Adhesive	50	10	60	60	20	70
Cohesive	10	70	20	10	50	10

Mixed	40	20	20	30	30	20

According to the obtained results, the µSBS of Panavia F2 cement was significantly higher than Dou-Link and TheraCem. Panavia F2 was used with ED primer as a self-etch primer [4]. ED primer simultaneously etches and primes the dentin surface with no acid application [4, 27]. Panavia F2 was containing 10-methacrylolyoxy-decyldihydrogen phosphate (10-MDP) in both ED primer and RC [23]. The Panavia F2 and HVGIC chemically bond probably due to the interactions between the dihydrogen phosphate group of 10-MDP from Panavia F2 and the calcium ions from the HVGIC matrix [23]. 10-MDP is the hydrophilic phosphate monomer that causes acidic decalcification and binding to calcium ions or amino groups of tooth structure and thus, increases resin diffusion and adhesion [28]. Moreover, there are acidic monomers in the ED primer with a pH of 2.4 [29] which shows better compatibility with the HVGIC matrix and leads to higher µSBS HVGIC [30].

The other investigated RC is TheraCem that is a self-adhesive RC with calcium silicate additives [31]. As indicated by the results, the µSBS of TheraCem was significantly lower than Panavia F2. The self-adhesive properties of TheraCem are provided by the acidic monomer of 10-MDP [32]. This functional monomer/10-MDP in Panavia F2 and TheraCem demineralizes and infiltrates the substrate of the tooth causing micromechanical retention [33-34]. However, high viscosity and low flowability of self-adhesive cements limit them to work only on the surface and thus, result in less infiltration to the substrate [35]. This finding is in agreement with the previous research in which it was shown that applying only self-adhesive RC does not form any hybrid layer or resin tag on the dentine surface [36] **Error! Reference source not found.**. However, the study by Zhang *et al.* [37] showed different finding where it was reported that one-step and two-step self-etching adhesive has no positive effect in improving the bond strength of resin composite to conventional glass ionomer.

A conventional resin composite, Dou-Link, was also investigated in this study. This material requires a separate etching and adhesive application step prior to cementation [38]. According to the obtained results, µSBS of Dou-link is lower than Panavia F2 but does not show a significant difference with TheraCem. This finding is in accordance with a previous study that showed higher bond strength between conventional GICs and etch-and-rinse systems compared to the self-etching adhesives [37]. Although applying phosphoric acid etching on teeth before bonding resin and restorative materials is well-studied previously [39], the use of phosphoric acid on GIC remains debatable. Some previous researches have reported that acid etching of GICs would improve the bond strength of resin composite by the formation of a hybrid-like layer [40]. They found that aggressive acid etching would form a rough and porous surface on GIC, leading to infiltration of the bonding resin [1, 34].

The failure mode analysis provides valuable insight into the bonding performance of the tested cements. The predominance of cohesive failures in the group in which Panavia F2 was used correlated with its higher microshear bond strength (µSBS) values, indicating strong adhesion and effective stress distribution within the material rather than at the bonding interface. This suggests that Panavia F2 forms a more reliable bond with the substrate. In contrast, Dou-Link and TheraCem exhibited a higher incidence of adhesive failures, implying weaker interfacial adhesion. This finding aligns with their lower µSBS values, highlighting potential limitations in their bonding efficacy. The adhesive failures suggest that the bond between these cements and the substrate may be more susceptible to stress concentration at the interface, possibly due to inferior chemical interaction or inadequate penetration into the substrate.

This study had several limitations. One limitation of this study was the relatively short duration of sample storage, which may not fully replicate longer-term clinical storage scenarios. While our experimental design focused on immediate post-treatment effects, extended storage conditions could reveal additional degradation patterns or functional changes relevant to real-world applications. Future studies should incorporate prolonged storage timelines (e.g., weeks or months) under varying conditions (e.g., temperature, preservatives) to better align with clinical practice. Despite this constraint, our findings provide critical foundational data for early-stage treatment. As another limitation, simulation of all clinical aspects is not possible in *in vitro* researches and thus, more future clinical experiments are necessary to predict the clinical aspects of the treatment.

Additionally, more studies are needed to evaluate the properties of the bonding between new HVGIC and RC, especially with saliva contamination.

Conclusion

Different types of HVGICs did not affect their μSBS to RCs. Among different RCs, Panavia F2 showed the highest μSBS to HVGICs. No significant difference in μSBS was observed for TheraCem and Dou-Link.

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Conflict of Interest

The authors deny any conflict of interest.

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