Effects of dentin surface treatments on shear bond strength of glass-ionomer cements

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Summary

Aim. **The aim of this in vitro study was to evaluate the effect of different surface treatments on shear bond strength of a conventional glass-ionomer cement (GIC) and a resin-modified glass-ionomer cement (RMGIC) to dentin.**
Materials and methods. **80 bovine permanent incisors were used. 40 cylindrical specimens of a GIC (Fuji IX GP Extra) and 40 cylindrical specimens of a RMGIC (Fuji II LC) were attached to the dentin. The teeth were then randomly assigned to 8 groups of equal size (n=10), 4 for every type of glass-ionomer cement, corresponding to type of dentin surface treatments. Group 1: GC Cavity Conditioner; Group 2: 37% phosphoric acid gel; Group 3: Clearfil SE Bond; Group 4: no dentin conditioning (control). The specimens were placed in a universal testing machine (Model 3343, Instron Corp., Canton, Mass., USA) and subsequently tested for shear bond strength (MPa).**
Results. **ANOVA showed the presence of significant differences among the various groups. Post hoc Tukey test showed different values of shear bond strength for Fuji IX GP Extra and for Fuji II LC. The different conditioners variably influence the adhesion of the glass-ionomer cements tested. Conclusions. RMGIC shear bond to dentin was higher than GIC. The use of a Self-etch adhesive system improved the shear bond strength values of RMGIC and lowered the shear bond strength values of GIC significantly.**

Key words: conventional glass-ionomer cement, resin-modified glass-ionomer cement, dentin pretreatment, phosphoric acid, polyacrylic acid, self-etch adhesives, shear bond strength test.

Introduction

Glass-ionomer cements (GICs) were developed and first presented by Wilson and Kent (1) in 1972: the goal was finding an ideal restorative material with physical properties similar to tooth structure, with adhesion to dentine/enamel, with resistance to degradation in the oral cavity (2) and with ability to release fluoride (3). GICs are able to bond chemically to enamel, dentin, plastics, and non-precious metals (4, 5); other potential advantages of GICs are adhesion in a wet substrate and the release of fluoride ions over long periods (6). Previous studies showed that enamel adjacent to glass-ionomer cements was less deeply and less frequently demineralised compared with that adjacent to non fluoridated materials (7, 8). Moreover, glass-ionomer cements were shown to release fluoride longer and at higher levels than fluoride-containing composites (9). The concept that glass-ionomers can act as rechargeable fluoride release devices has been proposed (10). Studies reported that the regular use of fluoride toothpastes can result in the absorption of fluoride into the glass-ionomer and that this fluoride can subsequently be released into the adjacent tooth structure (11, 12). Initial formulation of GICs underwent several modifications with the intent to improve handling and physical properties. A remarkable improvement of this class of material occurred with the introduction of the resin-modified glass-ionomer cement (RMGIC). This material is characterised by the addition of photo-activated metacrilate, and a small amount of resin, such as 2-HEMA or Bis-GMA, to the conventional glass-ionomer cement (GIC) (13, 14). Over the years many studies have been carried out to demonstrate that GICs and RMGICs bond naturally to the tooth surface (15, 16). Bond strengths are lower than composite resins bonded with appropriate bonding agents, but the durability of the bond appears greater (17). This may be attributed to the bioactive nature of the interface between the cement and the tooth (15, 18), which leads over time to a strong, durable bond formed by an ion-diffusion process (19). Many conditioning solutions, such as polyacrylic acid and phos-

phoric acid in different concentrations have been investigated as a pre-treatment to GICs and RMGICs, in order to improve their adhesion to the dentin surface (20-23). Dentin conditioning can act differently on GICs and RMGICs due to the presence of resin components that can infiltrate into the demineralised dentin and after polymerisation result in micromechanical retention (24).
The purpose of the present in vitro study was to evaluate the effect of different surface treatments on shear bond strength of a conventional glass-ionomer cement and a resin-modified glass-ionomer cement to dentin.

Materials and methods

In the present in vitro study, 80 bovine mandibular incisor were collected and randomly divided into 8 groups (n=10). Tissue remnants and debris were removed with periodontal curettes. The teeth were stored in 0.5% chloramine solution for one week and later in distilled water at 4º C. The teeth were embedded in self-cured acrylic resin, obtaining 2.0 cm large x 2.0 cm thick specimens. The teeth were randomly assigned to 8 groups (n=10) according to the dentin treatment received. The vestibular enamel was removed to obtain a flat surface of dentin. In all groups, a high-speed turbine attached to a device was used for standardised dentin preparation. The lingual dentin surface was exposed using a high-speed carbide rotary instrument (# H21L.314.014; Komet, Germany) under copious water irrigation. The surface was rinsed with water and gently air dried. Different types of surface treatments were employed: GC Cavity Conditioner, 37% phosphoric acid gel, Clearfil SE Bond, no dentin conditioning. 40 cylindrical specimens (4 mm diameter × 4 mm height) of a conventional glass-ionomer cement (Fuji IX GP Extra) and 40 cylindrical specimens (4 mm diameter × 4 mm height) of resin-modified glass-ionomer cement (Fuji II LC) were attached to the dentin. The capsules of glass-ionomer cements were activated and mixed according to the manufacturer's instructions. The prepared mixture was packed into a cylindrical plastic moulds (a diameter of 4 mm and a height of 4 mm) placed on the cut surface of the tooth, and packing them until they were full. In GIC specimens, there was a 6-min interval from the start of mixing to complete curing of the cement, but in RMGIC the specimens were immediately cured using a LED curing light in softstart-polymerisation mode (Celalux 2 High-Power LED curing-light, Voco GmbH, Cuxhaven, Germany) for 10 seconds at a light intensity of 1000 mW/cm^2. The tip of the light-curing unit was placed 1 mm above the surface of the cement. The teeth were then randomly assigned to 8 groups of equal size (n=10) corresponding to the type of dentin surface treatments and to the cylindrical specimen attached. Group 1: GC Cavity Conditioner (20% polyacrylic acid and 3% aluminum chloride hexahydrate) for 20 seconds, rinsed and gently air-dried plus Fuji IX GP Extra. Group 2: 37% phosphoric acid gel (Total Etch; Ivoclar Vivadent AG, Schaan, Liechtenstein) for 15 seconds, rinsed and gently air-dried plus Fuji IX GP Extra. Group 3: Clearfil SE Bond (Kuraray) applied according the Instruction for use plus Fuji IX GP Extra. Group 4: no dentin conditioning (control) plus Fuji IX GP Extra. Group 5: GC Cavity Conditioner (20% polyacrylic acid and 3% aluminum chloride hexahydrate) for 20 seconds, rinsed and gently air-dried plus Fuji II LC. Group 6: 37% phosphoric acid gel (Total Etch; Ivoclar Vivadent AG, Schaan, Liechtenstein) for 15 seconds, rinsed and gently air-dried plus Fuji II LC. Group 7: Clearfil SE Bond (Kuraray) plus Fuji II LC. Group 8: no dentin conditioning (control) plus Fuji II LC. Details of materials employed are presented in Table 1 and in Table 2.

Shear Bond Strength Testing

Specimens were stored in a solution of 0,1% (weight/volume) thymol for 24 hours and then were placed in a universal testing machine (Model 3343, Instron Corporation, Norwood, MA, USA) (Fig. 1). Specimens were secured in the lower jaw of the machine so that the bonded cylinder base was parallel to the shear force direction (Fig. 2). The shear bond strength was performed at 0.5 mm/minute until the sample ruptured. Specimens were stressed in an occluso-gingival direction at a crosshead speed of 1

Table 1. Glass-ionomer cements employed in the study.

Material	Type	Composition	Manufacturer	Batch Number
Fuji IX GP Extra in caps	GIC	Polyacrylic acid, Fluoro-alumino-silicate glass, Distilled water	GC Corporation, Tokyo, Japan	1102214
Fuji II LC in caps	RMGIC	2-hydroxyethyl methacrylate (HEMA), Urethane Dimethacrylate (UDMA), Polyacrylic acid, Fluoro alumino-silicate glass, Distilled water	GC Corporation, Tokyo, Japan	1103127

Table 2. Conditioners employed in the study.

Material	Composition	Manufacturer	Batch Number
GC Cavity Conditioner	20% polyacrylic acid, 3% aluminum chloride hexahydrate,distilled water	GC Corporation, Tokyo, Japan	1102171
Total Etch	phosphoric acid (37 % in H2O), thickeners and pigments	Ivoclar Vivadent AG, Schaan, Liechtenstein	P35844
Clearfil SE Bond	**Primer:** 10-MDP, HEMA, Hydrophilic dimethacrylate, CQ, N,N-Diethanol-p-toluidine, water. **Bond:** 10-MDP, Bis-GMA, Hydrophilic dimethacrylate, HEMA, CQ, N,N-Diethanol-p-toluidine, silanated colloidal silica.	Kuraray Medical, Sakazu, Okayama, Japan	Primer: 01040AA Bond: 01556AA

Figure 1. Universal testing machine Model 3343, Istrom (Universal testing machine, Model 3343, Instron Corporation, Norwood, MA, USA).

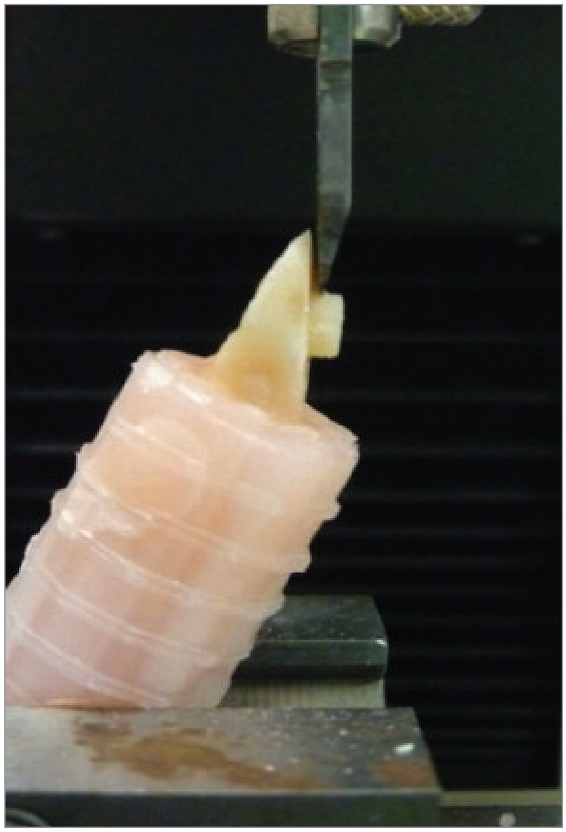

Figure 2. Universal testing machine Model 3343, Istrom (Universal testing machine, Model 3343, Instron Corporation, Norwood, MA, USA). The bonded cylinder base was parallel to the shear force direction.

mm/min (16-18). The maximum load necessary to debond was recorded in Newton (N) and calculated in MPa as a ratio of Newton to surface area of the cylinder. After the testing procedure, the fractured surfaces were examined with an optical microscope (Stereomicroscope SR, Zeiss, Oberkochen, Germany) at a magnification of 10x to determine failure modes and classified as adhesive failures, cohesive failures within the composite, or cohesive failures within the tooth (19). The adhesive remnant index (ARI) was used to assess the amount of adhesive left on the enamel surface (20). This scale ranges from 0 to 3. A score of 0 indicates no adhesive remaining on the tooth in the bonding area; 1 indicates mixed failure with less than half of the adhesive remaining on the tooth; 2 indicates mixed failure with more than half of the adhesive remaining on the tooth; and 3 indicates all adhesive remaining on the tooth. The ARI scores were used as a more complex method of defining bond failure site among the enamel, the adhesive, and the composite (25). Samples were thermocycled and debonded using a shear force with a crosshead speed of 0.5 mm/min. Shear bond strengths were determined using a Hounsfield Universal Testing machine, at a loading rate of 1 mm/min, using a knife edge placed 1 mm away from the interface. Loads at failure were converted to bond strengths by dividing by the contact areas of the cylinders. After mechanical failure, the fracture modes in all the specimens were evaluated under a stereomicroscope (Nikon; Japan) at ×20.

Statistical analysis

Statistical analysis was performed with Stata 9.0 software (Stata, College Station, Tex). Descriptive statistics, including the mean, standard deviation, median, and minimum and maximum values were calculated for all groups. Kolmogorov and Smirnov test was applied to assess normality of distributions. An analysis of variance (ANOVA) was applied to determine whether significant differences in debond values existed among the groups. The Tukey test was used as post-hoc. The chi-squared test was used to determine significant differences in the ARI scores among the different groups. Significance for all statistical tests was predetermined at $P<0.05$.

Results

Descriptive statistics of the shear bond strength (MPa) of the different groups are illustrated in Table 3 and in Figure. 3. ANOVA showed the presence of significant differences among the various groups ($P<0.0001$). Post hoc Tukey test showed that when testing Fuji IX GP Extra the application of cavity conditioner (Group 1) showed significantly higher shear bond strength than all other surface treatments ($P<0.05$). Moreover phosphoric acid gel application (Group 2) showed no significant difference in shear strength values than no conditioning control group (Group 4). Lowest values were reported when Clearfil

Table 3. Descriptive statistics of the different groups tested.

Groups	Material	Enamel pre-treatment	Mean	SD	Min	Mdn	Max	Tukey *
1	Fuji IX GP Extra	Cavity Conditioner	3.51	1.22	1.51	3.34	5.34	A
2	Fuji IX GP Extra	Ortophosphoric acid	1.86	1.10	0.18	1.11	5.02	B
3	Fuji IX GP Extra	Clearfil SE Bond	0.00	0.00	0.00	0.00	0.00	C
4	Fuji IX GP Extra	No conditioning	1.94	0.96	0.84	1.92	3.36	B
5	Fuji II LC	Cavity Conditioner	10.24	2.20	5.10	11.09	14.28	D
6	Fuji II LC	Ortophosphoric acid	6.53	1.90	2.08	6.30	11.23	E
7	Fuji II LC	Clearfil SE Bond	15.88	4.40	7.03	15.69	26.29	F
8	Fuji II LC	No conditioning	5.72	2.82	2.46	5.17	11.66	E

*: Tukey post hoc: Means with the same letters are not significantly different.

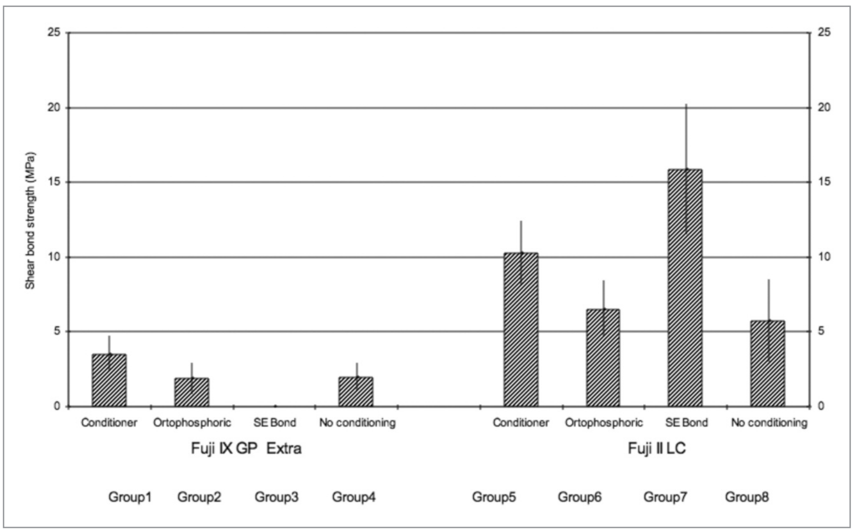

Figure 3. Mean shear bond strength and standard deviation of the different groups tested.

SE Bond was applied (Group 3). On the other hand, when testing Fuji II LC the highest shear bond strength values ($P<0.001$) were reported when Clearfil SE Bond was applied (Group 7). Significantly lower ($P<0.05$) values were reported when Cavity conditioner was applied (Group 5). Lowest shear strength values ($P<0.01$) were reported both when enamel was pretreated with phosphoric acid gel (Group 6) and when no conditioner was applied (Group 8). Overall, Fuji II LC showed significantly higher shear bond strength values than Fuji IX GP Extra ($P<0.0001$). When comparing ARI Score results of the different groups, no statistical difference was found in frequency distribution among various groups, that all showed a significant prevalence of ARI Score of “0” (Fig. 4).

Discussion

The adhesion of dental materials to dentin has been extensively investigated in the last decades in order to make it effective and durable, but due to dentin complexity this is an arduous procedure (26). Unlike enamel, dentin is a live, dynamic tissue that contains greater portion of water and organic material. It is connected to the pulp through the dentinal tubules, which extend from the pulp to the dentin-enamel junction. These tubules contain dentinal fluid that is responsible for the intrinsic humidity of this structure (26, 27). Different mechanical tests have been proposed to assess the bonding performance of restorative materials. Although it suffers criticism, shear testing has been widely used to evaluate the bonding ability of adhesive materials to dental structure (22, 28). Particularly regarding GICs, which present low bond strength, other tests may be difficult to apply (29). Previous studies have shown that typical shear bond strengths of glass-ionomer cements to dentine lie in the range 1–3 MPa, and rarely exceed 5 MPa (30, 31). The bond strength RMGICs to dentin have been reported as higher than that of conventional GICs (32). However, the exact mechanism of adhesion of this material is not completely established. Some SEM studies revealed the formation of tags at the dentin-cement interface resultant from the RMGIC polymer penetration into the dentinal tubules (21, 28, 33).

The application of surface-altering solutions to dentin prior to bonding with glass-ionomer cements has a long history (34, 35). The purpose of applying these solutions has been to increase the strength of the bond formed between the dentin surface and cement. For chemically-cured glass-ionomer cements, one of these first solutions used for this purpose was citric acid (36). Although 50% citric acid was commonly used as a dentin conditioning agent, it fell out of favour because it lacked biocompatibility (37), opened dentin tubules (38), and produced either no increase or decrease in bond strength (38, 39). Polyacrylic acid in various concentrations has also been suggested as a dentin conditioner prior to placement of chemically set glass-ionomer cement because Powis et al. (38) believed that it increases wettability of dentin surface and improves ion exchange with the cement. Although researchers have recommended its use in an attempt to maximise bond strength, suggested concentrations and application times have varied. Berry et al. (40) used SEM to evaluate dentin surfaces treated with number of conditioning solutions and concluded that a 5 second application of 40% polyacrylic acid produced the most ideal surface for bonding. However, Long et al. (41) found that a 30 second treatment with either 30% or 35% polyacrylic acid produced bond strengths that were significantly higher than those produced using 15%, 20%, 25% and 40% solutions. Although differences in opinion remain concerning application times and concentrations for polyacrylic acid, researchers continue to recommend its use as a dentin pre-treatment with chemically set glass-ionomer products. Polyacrylic acid is the most commonly used conditioner for conventional GICs because it is capable of cleansing the dentin surface

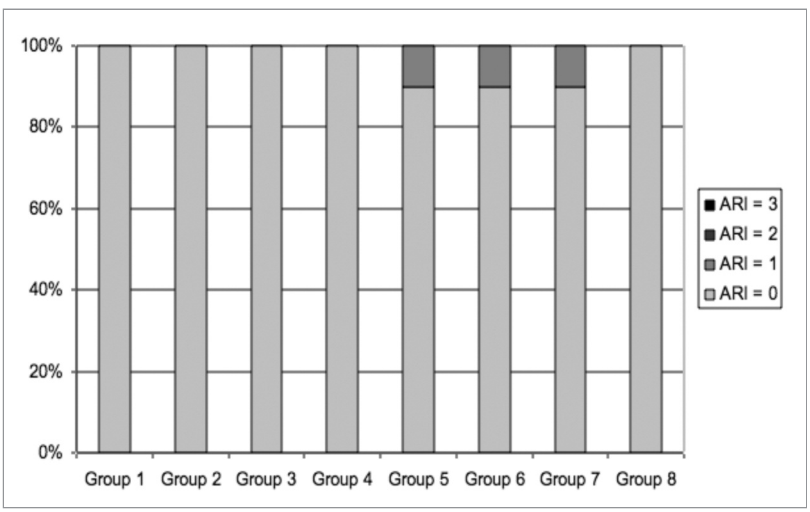

Figure 4. ARI Scores percentages of the different groups tested.

without completely unplugging the dentinal tubules. The increase in bonding efficiency resulting from conditioning can be attributed to: a cleansing effect which removes loose cutting debris following cavity preparation, a partial demineralisation effect which increases the surface area and creates microporosities and a chemical interaction of the polyalkenoic acid with residual hydroxyapatite. The auto-adhesion of glass-ionomer cements to tooth tissue has recently been elucidated to be two-fold in nature. Micromechanical interlocking is achieved by shallow hybridisation of the micro-porous, hydroxyapatite-coated collagen network . In this respect, glass-ionomer cements can be considered as adhering to tooth tissue through a kind of self-etch approach. As the second part of self-adhesion mechanism, true primary chemical bonding occurs through the formation of ionic bonds between the carboxyl groups of the polyalkenoic acid and calcium of hydroxyapatite that remained around the exposed surface collagen. The polyacrylic acid pre-treatment is much milder than a traditional phosphoric acid treatment, and the exposed collagen fibrils are not completely denuded of hydroxyapatite. The phosphoric acid treatment demineralised superficial dentin to a variable thickness of the order of several microns (depending on time of application) and the hydroxyapatite removal prevented formation of ion exchange in which the carboxyl groups of the cement interacted with calcium ions and phosphate from hydroxyapatite. Our research shows that the use of polyacrylic acid on dentin increases shear strength value of GIC whilst the use of another system for pre-treatment, phosphoric acid, shows no significant difference in shear strength values.

Dentin surface treatment remains a topic of research as new, resin-containing glass-ionomer products like visible light activated liners/ bases are introduced to the market. Prati et al. (23) evaluated the effects of nine different dentin surface treatments on the shear bond strength of Vitrabond to human dentin. They found that although many of the treatments significantly altered the dentin as observed using scanning electron microscopy, only neutral and acidic oxalate solutions significantly increased the bond strength. This finding implies that glass-ionomer products which contain resin may require dentin treatments that differ from those used with traditional glass-ionomer cements. It should not be surprising then that the dentin treatment used with recently developed visible light activated glass-ionomer restorative materials differs from those recommended for use with chemically set glass-ionomers forms. This is probably due to the liquid component that contains acrylic monomers: dentin treatment with dentin bonding primers rather than polyacrylic acid may be effective in maximising bond strengths. Prisma Universal Bond (30% hydroxyethyl methacrylate, 6% phosphonated penta-acrylate ester in ethanol) and similar primers that contain hydrophilic monomers facilitate wetting of dentin and enhance bonding between dentin and resin-containing materials (42). However, dentin treatment with polyacrylic acid is recommended by the manufacturer of Fuji II LC considering the fact that the liquid of Fuji II LC contains approximately 35% hydroxyethyl methacrylate (HEMA) (manufacturer's data).

Previous studies demonstrated a significant improvement in the bond strength of Fuji II LC after conditioning with polyacrylic acid (43-45). Pereira et al. (28) observed resin tag formation in dentin specimens pretreated with polyacrylic acid and restored with Fuji II LC. Fuji II LC contains HEMA which can facilitate an improvement in the wetting ability as well as suitable bonding (46). A hybrid-like layer was reported to form at the Fuji II LC/dentin interface when conditioning was carried out prior to application of this cement (44). According to many Authors phosphoric acid conditioning prior to RMGIC application was able to improve adhesion to dentin. The effective removal of the smear layer, exposure of collagen and opening of dentinal tubules promoted a better resin monomer (HEMA) penetration within the underlying dentin, thus creating a hybrid layer. Hybrid layer increases the surface energy and contributes to provide a better moisture of the dentin surface creating an interdiffusion zone between the cement and dentin matrix, which contributes to micromechanical retention, in addition to chemical adhesion to dentin (21, 22, 48). According to the previous research this study demonstrated the dentin bond strength of Fuji II LC using Clearfil SE Bond (contain hydrophilic monomers) was statistically higher than other treatments or no treatments (in the present study unconditioned specimens showed significantly lower bond strength results than all conditioned specimens). The better bonding performance of RMGICs compared to conventional GICs could be due to their expected dual mechanism of adhesion (32). Resin cements are composites of a resin matrix, such as Bis-GMA or urethane dimethacrylate, and fine inorganic particles as filler (48). HEMA is an example of a hydrophilic primer, used to improve the infiltration of adhesive monomers into demineralised dentin by wetting the surface of collagen fibres and maintaining the collagen network in an expanded state by stiffening the collagen fibres (49, 50). In addition, the increase in the bond strength can be attributed to the polymerisation of HEMA leaving of a film of polymerised material on the dentin surface (51, 52). Also, HEMA increases the infusion and impregnation of resin monomers into demineralised dentinal matrix. Thus, the interfacial hybrid zone formed by polymerized resins, including HEMA must have played an important role in enhancing the bonding of resin materials (53).

Conclusions

Within the limitations of this study and according to the methodology used in our study and the statistical analysis obtained, the following conclusions can be drawn:

- RMGIC showed significantly higher shear bond strength to dentin than GIC; this can be attributed to action of HEMA that can have played an important role in enhancing the bonding of RMGIC;
- the use of adhesive system, as Clearfil SE Bond, improved shear bond strength of RMGIC to dentin because it contains hydrophilic monomers that enhance bonding between dentin and RMGIC;
- the application of adhesive system, as Clearfil SE Bond, significantly lowered shear strength of GIC: therefore adhesive application is not recommended when using conventional glass-ionomer cements.

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