Minimally Invasive Dentistry: Bond Strength of Different Sealant and Filling Materials to Enamel

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Purpose: The purpose of this study was to evaluate the shear bond strength of different sealant and filling materials, used in minimally invasive dentistry, to human enamel.

Materials and Methods: Thirty-five sound extracted third molars were selected. The crowns were longitudinally sectioned, embedded in polystyrene resin, and grounded until a flat enamel surface was reached. The samples were assigned into seven groups (n = 10), according to the materials: G1-Fluoroshield; G2-Clinpro; G3-Dyract AP; G4-F2000; G5-Vitremer; G6-Fuji IX; G7-Vidrion F. All materials were applied according to the manufacturer's instructions. The samples were stored in distilled water at 37°C for 24 h and submitted to a shear bonding strength test in a universal testing machine with a crosshead speed of 0.5 mm/min. The failure sites were observed in Scanning Electron Microscopy (SEM). The data were submitted to ANOVA and Tukey's tests ($p < 0.05$).

Results: The mean values (MPa) of shear bond strength were for Fluroshield (25.92 ± 8.83), Vitremer (20.41 ± 13.34), Dyract AP (17.08 ± 6.38), Clinpro (12.82 ± 8.38), F2000 (8.71 ± 3.74), Fuji IX (7.64 ± 2.57), and Vidrion F (4.54 ± 2.11). Fluroshield resin sealant and Vitremer resin modified glass-ionomer showed statistically higher shear bond strength values than the conventional glass ionomer (GIC) cements. Clinpro and F2000 showed bond strength values with statistical difference only from Fluroshield. The failure mode varied among the groups. The majority of samples presented mixed failure.

Conclusion: FluroShield and Vitremer showed better performance of shear bond strength to enamel than conventional GIC.

Key words: shear bond strength, sealants, filling materials, resin modified glass-ionomer

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Occlusal caries now accounts for most of the lesions in children aged eight to 15 years. The majority of caries lesions in children are located in pit and fissures (Grande *et al*, 2000).

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This high incidence of occlusal caries in children is due to the capacity of deep pit and fissures to harbor bacterial and nutrients close of the dentin-enamel junction and, besides, to the difficulty or inability of the mechanical debridement of this area (Tandon et al, 1989). The high susceptibility of pit and fissures to carious attack and the rapid onset of the disease at this site soon after tooth eruption is reported by several researchers (Dirks, 1961; Hennon et al, 1969; Lewis and Hargreaves, 1975).

As a consequence of the increased understanding of the caries process and development of restorative materials, the concept of minimally invasive dentistry was introduced (Tyas *et al*, 2000).

Minimally invasive dentistry is a philosophy in which the goal of intervention is to conserve healthy tooth structure – not only by preventing disease from occurring and intercepting its progress but also by restoring fissure caries with maximum retention of sound tooth structure and sealing unaffected areas (Whitehouse, 2004; Erickson, 2004; Erickson *et a.*, 2003; White and Eakle, 2000).

Sealing pit and fissures is a proven method to prevent occlusal caries. (Kilpatrick et al, 1996; Poulsen et al, 2001; Pardi et al, 2003). According to the results of a systematic review of the literature, sealing with resin-based sealants is a recommended procedure to prevent caries of the occlusal surfaces of permanent molars (Uribe, 2004; Ahovuo-Saloranta *et al*, 2004).

In the same way, GIC has been used in conservative procedures as sealants or filling material in pediatric dentistry, and as atraumatic restorative treatment (ART) material. GIC bonds directly to teeth and releases fluoride, providing a potential effect of remineralization. Disadvantages of conventional GIC compared to resin materials are inferior mechanical properties, bond strength, tensile strength and fracture toughness. (Tyas *et al,* 2000)

Resin modification of GI cement (RMGICs) was designed to produce favorable physical properties similar to those of composite resins while retaining the basic features of the conventional GI cement. This goal was achieved by incorporating water-soluble resin monomers into an aqueous solution of polyacrylic acid. In this way the system undergoes polymerization of the resin monomer while the acid-base reaction continues simultaneously. RMGICs exhibit many advantages from both resin cements and glass-ionomer cements: they have better physical properties, are more aesthetic and less water-sensitive than conventional GIC. (Almuammar et al, 2001)

Another material, the polyacid-modified resin composite, so-called compomer, is composed by an anhydride with metacrylate groups. During light exposure the metacrylate groups start polymerizing and cause the material set. Over time, water diffuses into the material and converts the anhydride groups to polyacids capable of reacting through an acid-base reaction with filler particles. Due to this composition, the compomers can exhibit mechanical properties similar to those of resin composites, without losing the ability of fluoride releasing. (Almuammar *et al*, 2001; Schulze et al, 2003)

Therefore, in order to obtain long-term success with the sealant and the conservative restorations, an important condition is the presence of a satisfactory retention of the material to the enamel (Feigal, 1998). In the search for the best performance of materials, the shear bond strength is an important test in determining the retention degree of these materials in pits and fissure surfaces. (Castro and Galvão, 2004; Poulsen *et al,* 2001)

The purpose of this study was to evaluate the shear bond strength of different sealant and filling materials used in minimally invasive dentistry to human enamel. The null hypothesis tested was that all materials would present similar bond strength to the human enamel.

MATERIALS AND METHODS

Specimen preparation

Thirty-five extracted human third-molars were selected, cleaned, and stored in a 0.5% chloramine T solution for up two months after extraction. The roots of the teeth were sectioned off 1 mm under the cement enamel junction, and the crowns were sectioned in the mesio-distal direction using a double-face diamond saw (KG Sorensen, São Paulo, SP, Brazil). Then they were mounted on a ¾-inch-diameter PVC ring, parallel to the base of the ring. The rings were filled with self-curing polystyrene resin and the embedded specimens were ground on a water-cooled mechanical polisher (Minimet 1000, Buheler, UK Ltd, Lake Bluff, IL 60044 – USA) using 320-, 400-, and 600-grit silicon carbide abrasive paper (Carbimet Disc Set, #305178180, Buheler, UK Ltd, Lake Bluff, IL 60044 – USA) to expose a flat enamel area of at least 3 mm in diameter on the lingual, buccal, or palatine surfaces.

The specimens were randomly assigned into seven groups (n = 10). Before the surface treatment, the enamel surface was covered by an adhesive tape with a 3 mm-diameter hole (Fig 1a).

The materials used in this study and their components are described in Table 1. The groups received the following materials:

- **Group 1 (Fluroshield):** The enamel surface was etched using 37% phosphoric acid (H_3PO_4) gel for 30 secs, rinsed in water for 10 secs, and dried. Then a bipartite silicon ring mold (3 mm in diameter and 5 mm height – Fig 1a) was posi-

Table 1 Materials used in present study

Materials	Types	Composition	Manufacturers
Fluroshield	Resin sealant	Urethane modified Bis-GMA dimetacrylate; Barium aluminoboro-silicate glass (30 vol.%); Polymerizable dimetacrylate resin; Bis-GMA, Sodium fluoride; Dipentaerythritol pentaacrylate phosphate; Titanium dioxide; Silica amourphous.	Dentsply, Germany
Clinpro	Resin sealant	Bis-GMA; TEGDMA; Photo-initiator system; Camphorquinone; TBATBF; Butylatedhydroxytoluene; Silane treated amorphous silica; TiO_2; Rose Bengal sodium.	3M/ESPE, USA
Dyract AP	Compomer	Cetyllamine hydrofluoride acetone; UDM resin TCB resin; Polymerizable resins; Strontium fluoro-silicate glass; Strontium fluoride; Initiators/stabilizers.	Dentsply, DeTrey, Germany
Prime & Bond NT	Adhesive system	UDMA; PENTA; R5-62-1 resin; T-resin; D-resin; Butylated hydroxytoluene; Canphorquinone; 4-ethyl dimethyl aminobenzoate; Cetylamine hydroxyfluoride; Amorphous silica, Acetone	Dentsply, DeTrey Germany
F2000	Compomer	Fluoraluminosilicate glass; Colloidal silica; CDMA oligomer; GDMA; Hydrophilic polymer; Camphorquinone/amine.	3M/ESPE, USA
Single Bond	Adhesive system	BisGMA; HEMA; Dimethacrylates; Ethanol; Water; Photoinitiator system; Methacrylate functional copolymer of polyacrilic and polyitaconic acids.	3M/ESPE, USA
Vitremer	Resin-Modified glass- ionomer	Powder: fluoraluminosilicate glass, redox catalyst system, pigments Liquid: aqueous solution of a polycarboxylic acid modified with pedant methacrylate groups, Vitrebond copolymer, water, HEMA, photoinitiators. Primer: Vitrebond copolymer, HEMA, ethanol, photoinitiators.	3M/ESPE, USA
Fuji IX	Conventional Glass Ionomer	No manufacturers offered	GC Co, Japan
Vidrion F	Conventional Glass Ionomer	No manufacturers offered	SSWhite, Brazil

tioned over the treated enamel. The mold was filled with Fluroshield, in two increments and light cured for 20 secs, each increment, using Elipar tri-light unit (ESPE – America Co, Seefeld 82229 – Germany). Light intensity was periodically measured in the unit and ranged from 580 to 720 mW/cm^2.

- **Group 2 (Clinpro):** The same procedures as Group 1 were followed for Group 2, except for the application of Clinpro after acid etching.
- **Group 3 (Dyract AP):** The enamel surface was etched using a non-rinsed conditioner (NRC™ – 30% maleic acid, itaconic acid, methacrylate monomers) for 30 secs, and blot-dried. Prime and Bond NT was applied according to the manufacturer's instructions and light-cured for 10 secs. The silicon mold was positioned over the treated enamel. The mold was filled with Dyract AP in two increments and light cured for 20 secs, each increment using the Elipar tri-light unit.
- **Group 4 (F2000):** The enamel surface was etched using 37% phosphoric acid (H_3PO_4) gel for 30 secs, rinsed in water for 10 secs, and blot-dried. Single bond adhesive system was applied according to the manufacturer's instructions and light cured for 10 secs. The silicon mold was positioned over the treated enamel. The mold was filled with F2000 in two increments (Fig 1b) and light-cured for 20 secs, each increment using the Elipar tri-light unit.
- **Group 5 (Vitremer):** The enamel surface was treated with Vitremer Primer. The primer was

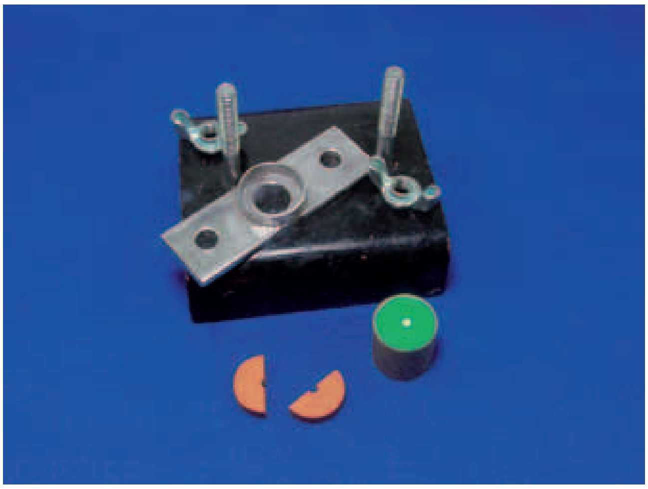

Fig 1a

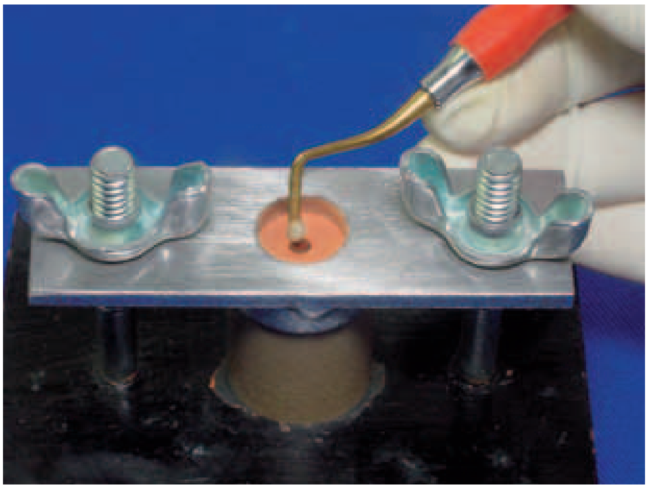

Fig 1b

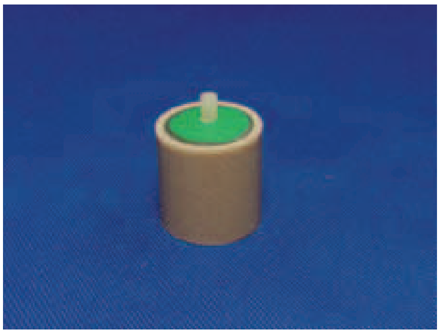

Fig 1c

Fig 1d

Fig 1a to 1d Shear bond strength testing method (a – Specimen preparation device: a1 – bipartite silicon ring mold; a2 – embedded specimen with enamel surface covered by an adhesive tape with a 3-mm-diameter hole; a3 – metallic mold and specimen holder; b – Filling of the silicon mold; c – Shear bond strength specimen (3 mm in diameter and 5 mm height); d – Shear bond strength test: d1 – material cylinder; d2 – stainless steel tape; d3 – Instron metallic specimen holder).

applied using a brush during 30 secs, air dried, and light-cured for 20 secs. The silicon mold was positioned over the treated enamel. Vitremer was manipulated according to manufacturer's instructions. The mold was filled with Vitremer in a single increment and light-cured for 20 secs using the Elipar tri-light unit.

- **Group 6 (Fuji IX):** The enamel surface was treated with polyacrylic acid conditioner (GC Conditioner) for 30 secs, rinsed in water for 10 secs, and dried. The silicon mold was positioned over the treated enamel. Fuji IX was manipulated according to manufacturer's instructions. The mold was filled with Fuji IX in a single increment.
- **Group 7 (Vidrion F):** The enamel surface was treated with polyacrylic acid conditioner (GC Conditioner) for 30 secs, rinsed in water for 10 secs, and dried. The silicon mold was positioned over the treated enamel. Vidrion F was manipulated according to manufacturer's instructions. The mold was filled with Vidrion F in a single increment.

The specimens (Fig 1c) were immersed in distilled water and stored for 24 hours at 37°C.

Bond strength test

Each specimen was submitted to the shear bond test in a universal testing machine (Instron – model 4411, Canton, MA 02021 – 1089 – USA). A stainless steel tape (5 mm in width and 10 cm in length) was placed around the material cylinder in close contact with the enamel surface, and the specimens were loaded to fail at a cross-head speed of 0.5 mm/min (Fig 1d) (Sinhoreti *et al,* 2001). Means and standard deviation were calculated with units expressed in MPa.

Table 2 Shear bond strength mean values (standard deviations) in MPa

Type of material	Material	Mean (SD)	
Resin sealant	Fluroshield	25.92 (8.8)	A
Resin modified glass ionomer	Vitremer	20.41 (13.3)	AB
Compomer	Dyract AP	17.08 (6.3)	ABC
Resin selant	Clinpro	12.82 (8.3)	BCD
Compomer	F2000	8.71 (3.7)	BCD
Conventional glass ionomer	Fuji IX	7.64 (2.5)	CD
Conventional glass ionomer	Vidrion F	4.54 (2.1)	D

Different letters show significant differences statistically ($p < 0.05$)

Statistical analysis

The data were submitted to ANOVA. Multiple comparison Tukey's test ($p < 0.05$) was chosen to check significant differences in means.

Failure type evaluation

All the specimens were observed in a stereomicroscope (Model XLT30 – Nova Optical Systems – Novo Tempo Co. e Participações LTDA, Piracicaba, SP, Brazil) at 25X magnification to classify the failure sites in:

1) adhesive failure (AF)
2) cohesive failure in the material (CM)
3) cohesive failure in the tooth (CT)
4) mixed – cohesive in the material and adhesive (CMA)
5) mixed – cohesive in the material, and in the tooth (CMT).

Three representative samples of each group were selected and observed in SEM (JEOL-JSM 5600LV, Tokyo, Japan).

RESULTS

The averages of the shear bond strength (SBS) for the seven groups are shown in Table 2. According to the statistical analysis, ANOVA and Tukey tests ($p < 0.05$), there was statistically significant difference among the groups ($p < 0.05$). The null hypothesis must be rejected. However, the materials showed no significant differences when compared in their respective types, except for the resin sealants. Fluroshield showed the highest strength, which was statistically superior to the strength of the Clinpro resin sealant. Fluroshield and Vitremer RMGIC showed significant difference from the Fuji IX and Vidrion F conventional GICs. Regarding the compomers, Dyract AP bond strength values were statistically different only from those of Vidrion F; and F2000 bond strength values were statistically different only from those of Fluroshield. The conventional GIC (Vidrion F) showed the lowest bond strength values, not differing from those of Clinpro, F2000, and Fuji IX.

The percentage of the failure types for all the groups are presented in Fig 2. The mixed failure (cohesive in the material and adhesive – Fig 3) was the most frequently observed in all the groups. The GI materials showed higher frequency of adhesive failure (Fig 4) when compared to the resin-based materials.

DISCUSSION

In this study, Fluroshield showed the highest mean values of shear bond strength of the seven groups evaluated. However, Fluroshield bond strength values were not statistically different from those of Vitremer and Dyract AP. The phosphoric acid etching produces porosities allowing better penetration of the resin monomers into the substrate (Wang et al, 1991). The etching also increases the surface area and energy enhancing the wettability of the sealant

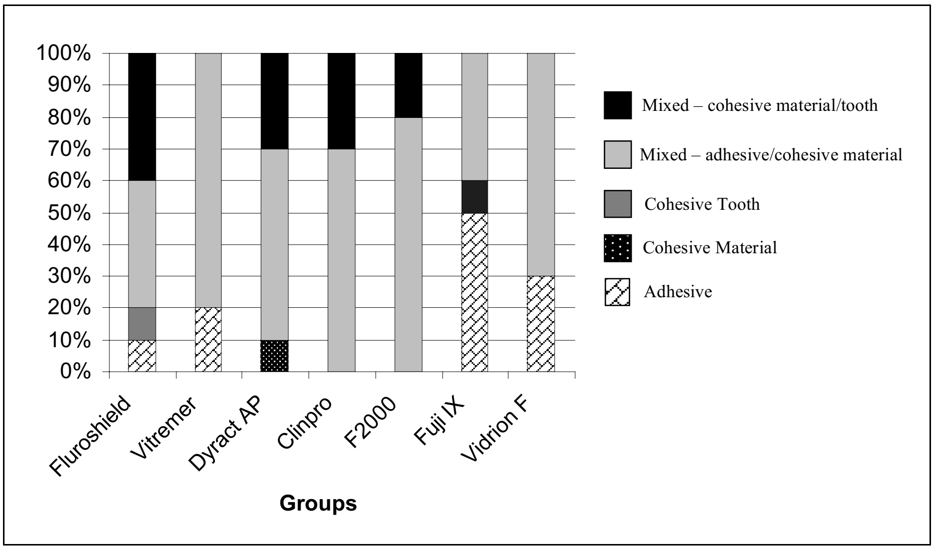

Fig 2 Percentage of failure type found after the shear bond strength test.

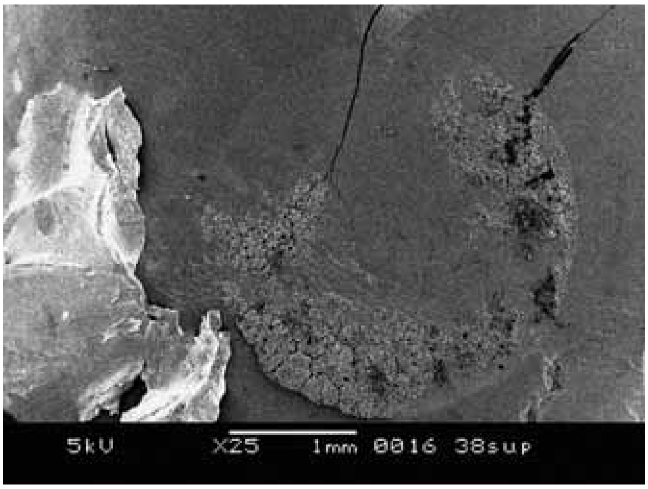

Fig 3 SEM photomicrography illustrating a mixed failure for Vitremer (A – adhesive; CM – cohesive in material).

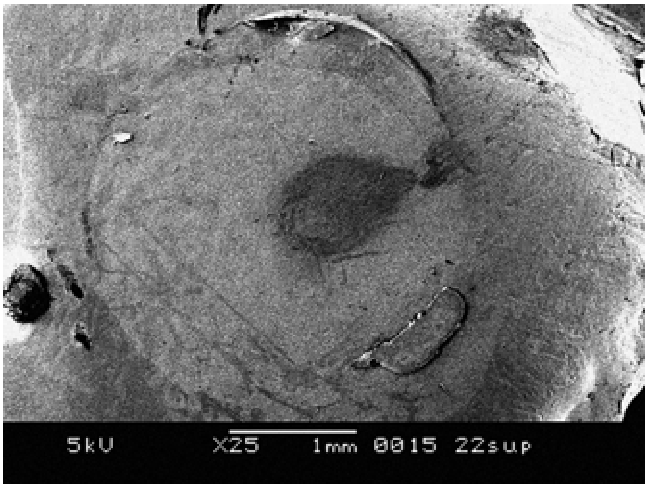

Fig 4 SEM photomicrography illustrating an adhesive failure for Vidrion F.

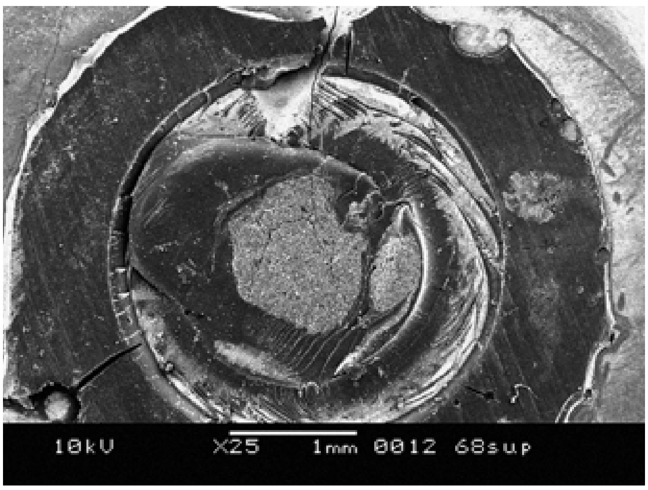

Fig 5 SEM photomicrography illustrating a mixed failure for Fluroshield (CT – cohesive in tooth; CM – cohesive in material).

material (Peutzfeld and Nielsen, 2004). The bonding is improved by the formation of a resin-infiltrated enamel layer and of resin tags in the subsurface of enamel (Peutzfeld and Nielsen, 2004; Irinoda *et al*, 2000). The efficacy of Bis-GMA materials used as fissure sealants has been documented (Forss and Seppa, 1990). The highest bond strength was confirmed by the failure mode analysis since Fluroshield sealant has showed the highest percentage of failure in dental structure, some cohesive in the tooth and some mixed (cohesive material/tooth – Fig 5).

Despite of the highest bond strength of Fluroshield, the resin sealant Clinpro showed statistically reduced bond strength. Although the restorative procedure is similar, the mechanical strength of these materials is different. The filler particles of Fluroshield are barium aluminoborosilicate glass (30 vol.%) and amorphous silica (2 vol.%) allowing better cohesive strength than Clinpro, which presents only silica in its composition.

The compomers fit in the minimally invasive dentistry since they have not only mechanical strength similar to resin composites but also fluoride release. Likewise the resin composites, the compomers are not able to bond to dentin and enamel, requiring the enamel etching and use of bonding system as composite resins.

In this study, the compomers Dyract AP and F2000 were not statistically different, presenting similar bond strength and similar failure mode. Despite of that, they follow different bonding procedures. F2000 uses conventional phosphoric acid etching followed by single bond adhesive system application. This procedure results in substantial penetration of the resin into the microporosities created by the etching and is believed responsible for the strength and durability of the bond (Glasspoole, Erickson & Davidson, 2001; Abate *et al.,* 1997). In Dyract AP group, NRC™ was applied to enamel followed by the bonding system Prime & Bond NT. The NRC™ etching, as that of phosphoric acid, increases the surface energy, porosity and microretention (Peutzfeld and Nielsen, 2004), promoting more ionic interactions between the material components and the enamel (Bishara *et al,* 2001; McLean et al, 1994). The low viscosity of adhesive system allows its penetration in the etched enamel. Dyract AP was claimed to bond chemically to enamel and dentin by ionic interaction between the hydrophilic phosphate group on the PENTA from adhesive system and the calcium ions from the dental hydroxyapatite. However, the chemical bonding to tooth surface is weak and does not contribute to maintain the long-term retention (van Noort, 2002).

Vitremer was used in this study for its anti-caries and cariostatic effects (Pardi *et al*, 2003; de Luca-Fraga and Pimenta, 2001; Pereira *et al*, 2001). The addition of resin to the GIC has significantly improved many of their properties. The advantages of GIr cements, such as the ability to bond to dentin and enamel and to release fluoride, are combined with a prolonged working time and rapid set, once irradiated with visible light (van Noort, 2002).

The results showed higher bond strength for Vitremer than Fuji IX and Vidrion F. The values were comparable to those obtained for the resin sealant Fluroshield. Vitremer Primer has acid monomers able to etch the substrate and enhance the micromechanical retention (Glasspoole et al, 2002). Additionally, there is a chemical adhesion: the polyacrylate ions react with the apatite structure or bond directly to the calcium in the apatite. Other studies have confirmed the good retention rate of Vitremer (Pardi *et al*, 2003; de Luca-Fraga & Pimenta, 2001; Pereira *et al.*, 2001).

Regarding the failure mode analysis, the most frequent failure mode was mixed (adhesive/cohesive in material – Fig 3) for almost all the groups, especially for Vitremer and F2000. This failure mode is frequently observed in shear bond strength tests, and it is related to the stress distribution during the test (Kitasako *et al,* 1995).

The Conventional GIC (Fuji IX and Vidrion F) showed the lowest shear bond strength mean. However, Fuji IX bond strength values were not statistically different from those of Dyract AP, Clinpro, F2000, and Vidrion F; and Vidrion F bond strength values were not statistically different from those of Clinpro, F2000, and Fuji IX. The reduced bond strength occurred because the cohesive strength of the conventional GIC is lower than the other materials. In addition, the bond to tooth surface is essentially chemical, with interaction between carboxylic groups from polyacid and calcium from enamel (Burgess *et al,* 1996), which is also responsible for the lowest shear bond strength observed for these materials.

The results herein are supported by Poulsen *et al* (2001). According to these authors, the major problem of conventional GI sealants is that they generally exhibit poor retention rates. On the other hand, Pardi *et al* (2003) observed in a clinical five-year evaluation that the caries preventive effect of GI sealants remained even after sealant loss, which has been attributed due to the continuous fluoride release from remaining material on the bottom of the fissures (Peters and Roeters, 1994; Forss and Seppa, 1990). Despite the low shear bond strength of some materials used as pit and sealants, and the need of material retention to inhibit caries (Poulsen *et al,* 2001), the fluoride release may reduce lesion initiation and progression (Croll, 1990), due to the incorporation of fluoride into the enamel adjacent to the material and the cavosurface enamel, resulting in a reduction in enamel solubility (Jensen et al, 1990), which could be a mechanism of enhanced caries resistance (Hicks et al, 2000).

In addition, it should be considered that GIC sealants could be used as temporary sealants in the early eruption stage molars, because of the difficulty of adequately isolating the tooth during the application of the sealant. In this case, the GIC retention is less sensitive to moisture than resin-based sealants retention (Pardi *et al*, 2003).

Failure analysis of the conventional GICs revealed their brittle characteristic once several specimens showed some cohesive fracture in the material, supporting the results of shear bond strength test. In this analysis, high frequency of adhesive failures (Fig 4) was also observed, confirming the reduced bond strength of the conventional GICs, corroborating with Cortes *et al* (1993).

Minimally invasive dentistry, such as sealing, ART and conservative restorations, can modify the patient caries risk, preventing and reverting caries lesion progress. The cost of these procedures is much lower than conventional restorative treatment, and the greatest benefit is found in the conservation of dental integrity.

CONCLUSION

Resin sealant (FluroShield) and RMGIC (Vitremer) showed the best performance of shear bond strength to tooth structure as confirmed by the SEM analysis.

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Clinical relevance: Fluroshield show higher shear bond strength than Conventional Glass Ionomer Cements, what could be related with more retention of this material on tooth structure.