Mechanical Properties and Bond Strength of Glass-ionomer Cements

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Purpose: The objective of this study was to evaluate the mechanical properties and bond strength of glass-ionomer cements (GICs) and resin-modified GICs (RM-GICs) that are indicated as restorative materials for the Atraumatic Restorative Treatment (ART) technique.

Materials and Methods: Fifteen disk specimens for the diametral tensile strength (DTS) test and fifteen cylindrical specimens for the compressive strength (CS) test were made of each GIC: Ketac-Fil, Ketac-Molar (ESPE), Fuji IX and Fuji PLUS (GC). Forty human molars were sectioned and embedded in resin with either buccal or lingual surfaces exposed for the tensile bond strength (TBS) test. The surface was ground until a flattened area of enamel or dentin was obtained. After conditioning, inverted truncated cones of GICs were prepared on the flat tooth surfaces. The powder:liquid ratio of Fuji PLUS was adjusted for restorative purposes. Prior to testing, specimens were stored for 24 h (TBS test) and for 1 h, 24 h, and 7 days (CS and DTS tests) in deionized water at 37°C. They were then loaded at a crosshead speed of 1.0 mm/min for CS and 0.5 mm/min for DTS and TBS tests until failure occurred. The data were submitted to two-way ANOVA at 0.05 level of significance, followed by a Tukey-Kramer test for multiple comparisons.

Results: The mean CS values ranged from 90.27 to 170.73 MPa and DTS means from 6.21 to 22.32, with test periods from 1 h to 7 days. The means for TBS ranged from 4.90 to 11.36 MPa and from 2.52 to 5.55 MPa in enamel and dentin, respectively. No differences were found between materials with the CS test except at 1 hour. The resin-modified GIC (RM-GIC) had the highest DTS, with no changes between the test periods, and the highest TBS for both enamel and dentin.

Conclusion: Among the GICs tested, RM-GIC showed higher values of DTS and TBS.

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Since the development of glass-ionomer cement (GIC) by Wilson and Kent at the beginning of the 70 s, many changes in the original formulation have been made in order to increase its clinical applica-

tions. This is understandable, because this material has advantageous properties such as a coefficient of thermal expansion similar to that of enamel and dentin,[60] physico-chemical adhesion,[11,21,36] biocompatibility,[6,56] and fluoride ion release.[10,40] The fluoride may decrease caries recurrence and may act in the remineralization of decayed dentin.[17]

This cement might be useful in the Atraumatic Restorative Treatment (ART) technique.[30,41] The main objectives of ART are to preserve dental structure and provide preventive and curative care to needy populations.[19,20] This technique consists of cavity preparation limited to removal of unsupported enamel and dentin and caries excavation only by means of hand instruments. The tooth restoration is completed with the placement of a conventional GIC.[30]

At the beginning of the 90 s, condensable glass-ionomer cements, more properly denoted as high-viscosity GIC,[18] were developed. When compared with conventional GIC, these new materials kept their anti-cariogenic properties but showed superior abrasion resistance.[46] Another route to obtain the above-mentioned improvements is to use a chemically-cured resin-modified glass-ionomer cement (RM-GIC) that offers improved properties attributable to resin cross linking of the polyalkenoate chains and polymeric hydrogels.[39,43] The original application of this type of cement was for cementation of prosthetic appliances. In order to adapt it for use in the ART technique, the powder:liquid ratio was increased by the manufacturer to obtain restorative consistency.[15]

Ewoldsen et al[15] have tested the mechanical and adhesive properties of one high-viscosity GIC and one chemically-cured RM-GIC suitable for the ART technique. They used the shear bond strength to test adhesive properties. The consistency of the material they used was thinner than the usual consistency for restorations.

The powder:liquid ratio of glass-ionomer cements has a definite influence on the mechanical properties[27,61] and bond strength of these materials.[15] It would be important to test chemically cured RM-GIC in a proper restorative consistency and high-viscosity GIC in comparison with conventional GICs regarding their mechanical properties and tensile bond strength. The aim of this study was to compare mechanical properties and bond strength of high-viscosity GICs and a chemically cured RM-GIC with an increased powder:liquid ratio (Table 1) to provide a basis for the selection of GICs suitable for ART technique.

MATERIALS AND METHODS

The four chemically-cured glass-ionomer cements tested in this study are listed in Table 1.

Mechanical Tests

To evaluate compressive (CS) and diametral tensile strengths (DTS), the ANSI/ADA Specifications Nos. 27[42] and 66[2] were followed. The ratios of powder to liquid were used according to manufacturers' instructions for all materials, except in the case of Fuji PLUS, for which the ratio was increased to gain a restorative consistency (Table 1). The material necessary to make each specimen was weighed in a precision balance and mixed with a plastic spatula on impermeable paper.

The specimens were made at a room temperature of 23°C ± 2°C and relative air humidity of 50% ± 10%, as recommended by ANSI/ADA specification.[2] After mixing, the materials were inserted with a Centrix syringe (Caulk Dentsply, Rio de Janeiro, Brazil) into metallic matrices, previously coated with a thin layer of petroleum jelly. The insertion was done slowly to adapt the material into the matrix and avoid bubble formation.

In accordance with ANSI/ADA specifications Nos. 27[42] and 66,[2] five specimens were prepared for each material for each of three periods of time: 1 h, 24 h, and 7 days. The cylinder dimensions were 6.0 mm diameter x 12.0 mm for the CS test and 6.0 mm diameter x 3.0 mm for the DTS test. After setting reaction (20 min), the specimens were removed from the matrices and immersed in deionized water at 37°C for the entire respective storage period.

The specimens were tested in a universal testing machine (Kratos, model K500/2000, Kratos, São Paulo, SP, Brazil) at a crosshead speed of 1.0 mm/min for CS and 0.5 mm/min for the DTS test. For the CS test, the specimens were placed in a vertical position, with force incident on the long axis, while for the DTS test, the specimens were positioned horizontally in their diametral direction.

Bond Strength Test

Forty extracted human molars without cavities were stored in saline solution with a thymol crystal at room temperature. These teeth were sectioned in a mesio-distal direction, and after protecting the pulp

Table 1 Materials, manufacturers, GIC classification, powder: liquid ratio suggested by the manufacturer or used in the present study, and batch numbers

Product	Manufacturer	GIC Classification	Manufacturer P:L ratio	P:L ratio used in this study	Batch number
Ketac-Fil Plus	ESPE, Seefeld, Germany	Conventional (traditional)	3.2:1.0	3.2:1.0	FW0042242 FW0041164
Ketac-Molar	ESPE	Conventional with high viscosity	3.0:1.0	3.0:1.0	FW0042242 FW0041164
Fuji IX	GC, Tokyo, Japan	Conventional with high viscosity	3.6:1.0	3.6:1.0	071071
Fuji PLUS	Fuji	Resin-modified	2.0:1.0	3.8:1.0	250671

Table 2 Materials and respective conditioners, components, and procedures used to chemically treat surfaces

Material	Conditioner	Component	Procedure
Fuji PLUS	Fuji PLUS Conditioner	Citric acid 5% to 10% and ferric chloride 1% to 3%	Applied actively for 20 s, washed and dried with absorbent paper
Fuji IX	Fuji IX liquid	Polyacrylic acid 39% and tartaric acid 11%	Applied with cotton pellet moistened with water for 20 s, cleaned with cotton pellet, moistened with water 3 times, and dried with a fresh cotton pellet
Ketac Molar	Durelon liquid	Polyacrylic acid 40%	Applied actively for 10 s, washed, and dried with absorbent paper
Ketac-Fil	Durelon liquid	Polyacrylic acid 40%	Applied actively for 10 s, washed, and dried with absorbent paper

area, each was embedded horizontally in an acry-lic resin with buccal or lingual surfaces exposed. These surfaces were wet ground using 320- and 600-grit silicon carbide paper disks, so that flattened enamel and dentin surfaces were obtained.

The specimens were divided into eight groups, each containing five specimens, for each GIC to be tested both on enamel and dentin surfaces. An adhesive tape with a 3-mm-diameter hole was firmly attached to these surfaces to restrict the adhesive areas that were conditioned. When using Fuji PLUS, the surfaces were treated with Fuji PLUS Conditioner. With Fuji IX, the surfaces were treated with Fuji IX liquid according to manufacturer's instructions. In the Ketac-Molar and Ketac-Fil groups, the corresponding tooth surfaces were treated with Durelon liquid (ESPE, Seefeld, Germany) (Table 2).

The specimens were positioned in a metallic device that fixed them during preparation. Inverted truncated cones of GICs were prepared by means of a split polytetrafluoroethylene mold positioned against the flat tooth surfaces. Powder and liquid of each material were weighed in a precision balance before hand mixing. All materials were manipulated according to manufacturers' instructions (except Fuji PLUS, as above) as shown in Table 2. All materials were syringed into the specimen molds. The polytetrafluoroethylene mold was filled with GIC and the exposed material was covered with a Mylar tape under moderate finger pressure for 1 min.

After setting reaction (about 20 min), the specimens were separated from the mold. The GICs were protected by clear nail varnish, and the specimens were stored at 37°C in deionized water. After 24 h, the specimens were placed in an appropriate loading jig (Fig 1) to be submitted to a tensile bond strength test in a universal testing machine (Kratos, model K500/2000, São Paulo, SP, Brazil) at a crosshead speed of 0.5 mm/min. The force required to fracture the specimen was recorded and

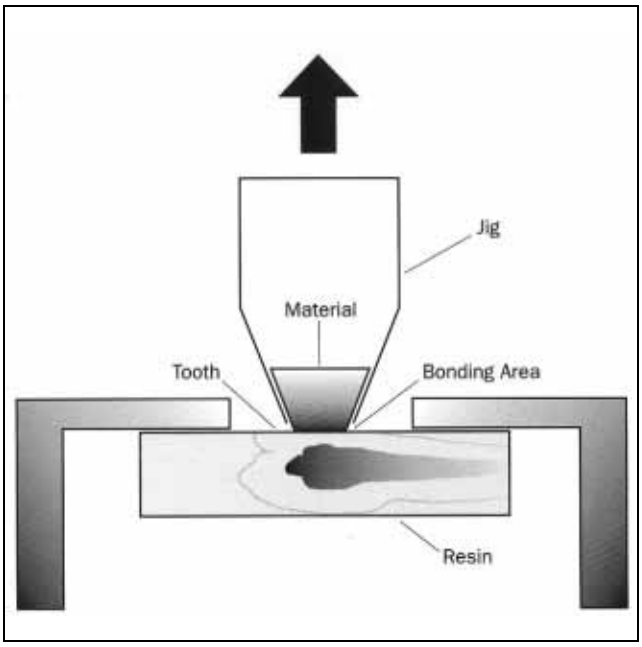

Fig 1 Specimen positioned in an appropriate device for tensile bond strength test procedure.

the bond strength was calculated and expressed in MPa. The mode of failure was noted after a visual examination using a light microscope under 40X magnification. Failures were recorded as adhesive (those which occurred between the GIC and tooth structure), cohesive (those which occurred within the GIC or tooth structure), or mixed (combination of adhesive and cohesive).

Statistical Analysis

The test data were analyzed for significant differences by two-way ANOVA and, where appropriate, by Tukey-Kramer tests for individual comparison with a 0.05 level of significance.

RESULTS

The CS and DTS test results for the GICs are shown in Tables 3 and 4. For the statistical analysis, a pre-set level of statistical significance was set at $p < 0.05$. For the CS (Table 3), the two-factor analysis of variance showed differences among materials, storage time, and interaction. In the DTS test (Table 4), the two-way analysis of variance detected differences among materials, storage time, and interaction. For the TBS test (Table 5), the two-way ANOVA detected differences among materials, tooth structure, and interactions.

Since the two-way ANOVA showed significant interactions in all three tests, it was not possible to determine significant main effects (for materials or time; and for materials or substrate) with the Tukey-Kramer test. Therefore, it was only possible to search for differences within rows or columns in Tables 3 to 5. No differences in compressive strength were observed between Fuji PLUS, Fuji IX, and Ketac-Molar for the 1 h period. After periods of 24 h and 7 days, no significant differences were observed between the materials. Significant differences in CS were also observed when comparisons were done between different water storage periods for each material. Fuji PLUS, Fuji IX, and Ketac-Fil had the highest CS after 24 h and 7 days, whereas for the Ketac-Molar group, no statistically significant differences were observed between test periods.

No significant differences were observed in DTS between the two high-viscosity GICs (Fuji IX and Ketac-Molar) tested for 1 h. On the other hand, Fuji PLUS was statistically higher in the DTS test, followed by high-viscosity GICs (Fuji IX and Ketac-Molar). At 24 h, Fuji PLUS presented the highest DTS values, and no statistically significant differences were observed among other groups. At 7 days, the DTS of Fuji PLUS was superior to that of the other materials, which all fell into the same statistical group. Analyzing the influence of the water storage time on DTS, it was found that Fuji PLUS was not influenced by water storage. In contrast, all materials showed an increase in DTS between 1 h and 24 h, which remained stable up to 7 days.

Tensile bond strength data are shown in Table 5. The RM-GIC, Fuji PLUS, showed the highest bond strength to enamel and dentin compared with the other GICs ($p < 0.05$). Fuji PLUS, Ketac-Molar and Ketac-Fil had higher bond strength to enamel than dentin ($p < 0.01$). No statistically significant difference was observed in the bond strength of Fuji IX to enamel and dentin. All failures found were either cohesive within the GIC or mixed. The majority of cohesive failures was found in the Fuji PLUS group.

DISCUSSION

Compressive and tensile stress tests are used in dentistry for laboratory simulation of the stress that may result from forces applied clinically to a re-

Table 3 Mean compressive strength of GIC in MPa and standard deviations (SD)

Product	1 h	24 h	7 days
Fuji PLUS	122.65 (4.24) $^{A\,1}$	136.32 (6.63) $^{C\,1\,2}$	157.54 (16.86) $^{D\,2}$
Fuji IX	132.36 (9.82) $^{A\,3}$	152.41 (14.02) $^{C\,3\,4}$	170.73 (3.98) $^{D\,4}$
Ketac-Molar	127.78 (7.44) $^{A\,5}$	149.18 (15.01) $^{C\,5}$	146.38 (11.91) $^{D\,5}$
Ketac-Fil	90.27 (4.05) $^{B\,6}$	146.28 (11.96) $^{C\,7}$	156.43 (20.57) $^{D\,7}$

Results designated with the same superscript are not statistically different ($p < 0.05$).
Letters are for comparisons between materials; numbers are for comparisons between different times.

Table 4 Mean diametral tensile strength values of GIC in MPa and standard deviations (SD)

Product	1 h	24 h	7 days
Fuji PLUS	22.32 (1.38) $^{A\,1}$	21.96 (0.80) $^{E\,1}$	21.78 (0.25) $^{G\,1}$
Fuji IX	8.53 (1.10) $^{B\,2}$	12.56 (1.54) $^{D\,3}$	10.64 (0.20) $^{F\,3}$
Ketac-Molar	8.65 (0.85) $^{B\,4}$	11.13 (0.94) $^{D\,5}$	10.76 (0.68) $^{F\,5}$
Ketac-Fil	6.21 (0.37) $^{C\,6}$	11.29 (0.66) $^{D\,7}$	12.86 (1.12) $^{H\,7}$

Results designated with the same superscript are not statistically different ($p < 0.05$).
Letters are for comparisons between materials; numbers are for comparisons between different times.

storative material.[46] Compressive stresses contri-bute to fracture failure through masticatory forces,[3] although an exact critical value is unknown.[9] A British specification established that the minimum value necessary to resist the masticatory forces in the posterior teeth would be 125 MPa, while some authors[58] believe that this value should be 100 MPa in primary dentition. Evaluations of compressive strength have shown that conventional GIC presents results superior to RM-GIC.[15] However, other studies have shown that RM-GIC has a compres-sive strength higher than that of conventional GIC.[7,31,35,37] In contrast, the different materials in this study did not show significant differences when compressive strength was evaluated.

Table 5 Mean tensile bond strength of GIC in MPa and standard deviations (SD)

Product	Enamel	Dentin
Fuji PLUS	11.36 (2.04) $^{A\,1}$	5.55 (0.94) $^{C\,2}$
Fuji IX	5.00 (1.59) $^{B\,1}$	3.79 (0.91) $^{D\,1}$
Ketac-Molar	5.31 (0.71) $^{B\,1}$	3.08 (1.03) $^{D\,2}$
Ketac-Fil	4.90 (1.00) $^{B\,1}$	2.52 (1.10) $^{D\,2}$

Results designated with the same superscript are not statistically different ($p < 0.05$).
Letters are for comparisons between materials; numbers are for comparisons between different substrates.

The diametral tensile strength test (DTS) is a critical requirement, because many clinical failures are due to tensile stress. In this study, the highest values were observed with RM-GIC. Similar results have been previously reported.[8,15,31,33] A probable explanation for this is that resin addition is an important modification to GICs[25] compared to conventional materials,[22,45] leading to improved mechanical properties. The addition of HEMA could result in a less-brittle material, in which case the specimen would deform before failure or fracture into more than two equal pieces. In this study, the specimens always fractured into two equal pieces, which shows that DTS was really tested.

The evaluation period of 1 h is justified because of the initial cure of GICs, while 24 h represents the second reaction phase, and the 7-day period allows

evaluation of whether the material gained strength with time.[9] It was evident in this study that by 24 h after the beginning of the curing reaction, most of the cements had not reached maximum strength. Some materials, however, can behave in a different manner with time. Many studies report an increase of DTS and CS with time, as occurred in this study, but the DTS of RM-GIC Fuji PLUS was not influenced by time (Table 4).

Physical properties of GIC are mostly influenced by the powder:liquid ratio.[5,12,15,32,57] The increase of powder content can decrease translucency, decrease the working and curing time,[12] and increase CS and DTS.[60] Additionally, the high viscosity is an important characteristic for the ART technique because it makes handling the material easier.[51]

Many different parameters can affect laboratory test values, and unfortunately one of them is a lack of test standardization. Different strength values reported in this study in comparison with Ewoldsen[15] can be attributed to differences in methodology. The specimens of the two studies had different dimensions. Since many articles published did not follow ANSI/ADA specifications, it is difficult to compare the results of this study with other studies. Thus, care must be taken when comparing the results of studies with different methodologies.[28]

Several factors can influence the bond strength, one of which is the type of dental substrate. Theoretical considerations and results of experiments show that enamel is much more susceptible to adhesion than dentin.[4] Enamel has a surface that is essentially homogeneous, dense, and chiefly composed of hydroxyapatite, which possesses high surface energy. Dentin has a heterogeneous surface, containing dental tubules that contain odonto-plastic processes, consists of approximately 30% volume organic matter, and consequently has low surface energy.[4] This could explain the differences found in the bond strengths between enamel and dentin for Fuji PLUS, Ketac-Molar and Ketac-Fil. For Fuji IX, this difference was not statistically significant. Ewoldsen et al[15] and Friedl et al[22] also did not observe significant differences when testing bond strength of this material.

Another factor that could influence the bond strength is the substance applied to the dental substrate.[48] Polyacrylic acid, one of the agents used in this study, removes the smear layer,[16] thus exposing the underlying dentin, increasing the contact area, facilitating the wetting of the surface, and pre-activating the calcium and phosphate ions of the dentin, making them more available for ionic reaction with the cement.[14,39,47,59] This mechanism achieves an intimate intermolecular contact between the adhesive cement and the tooth substrate.[14,47,59]

Fuji PLUS conditioner can modify the smear layer to promote the penetration of HEMA and other resinous monomers inside the tubules, which also results in micromechanical retention.[1,16,23,29] The mixed solution of citric acid and ferric chloride solution, present in Fuji PLUS conditioner, increases bond strength of GIC by removing smear layer without denaturing the dentin collagen.[47,54]

The highest bond strength values observed with RM-GICs in relation to those of the conventional GICs are commonly described in literature.[7,13,15,38,45,52] The explanations for this include the possibility of the formation of a hybrid layer, the development of the best wetting of the dentin by the HEMA contained in the RM-GIC,[16,23] its better mechanical properties,[23] and perhaps also the individual composition of each material.[24,55]

No statistically significant differences were observed in TBS between the high-viscosity cements (Fuji IX and Ketac-Molar) and the traditional cement (Ketac-Fil). The addition of 5% lyophilic polyacrylic acid to the powder and the finer glass particle size distribution[18] improved the mechanical properties of these cements mainly in the first hours, but it did not seem to improve the union with the dental structure.

The presence of cohesive and mixed failures, both in enamel and dentin, means that bond strength values represent only the tensile bond strength of the cement rather than the strength of the tooth-cement interface. This type of failure has been commonly reported in the literature when GICs are used in tests of bond strength.[1,7,13,23,26,34,38,44,47,49,50,53,55] It was intriguing that to the naked eye, many of the specimens seemingly showed truly adhesive failure. However, when examined under 40X, it was observed that the surfaces were covered with a fine layer of GIC, suggesting cohesive failure close to the tooth-cement interface.

CONCLUSIONS

The RM-GIC with restorative consistency exhibited higher DTS and TBS both to enamel and dentin than did other types of GIC tested. The CS test showed similar results for restorative-consistency RM-GIC and conventional GIC or high-viscosity GICs.

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