Qualitative evaluation of the adesive interface between lithium disilicate, luting composite and natural tooth

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Summary

Aim of this work was to qualitatively evaluate the interface between tooth, luting composite and lithium disilicate surface using a scanning electron microscope (SEM). An extracted restoration-free human molar was stored in physiological solution until it was embedded in an autopolimerysing acrylic resin. A standard preparation for overlay was completed and after preparation an anatomic overlay was waxed on the tooth and then hot pressed using lithium disilicate ceramic. After cementation the sample was dissected and the section was analysed using an Automatic Micromet (Remet s.a.s) and the section was analyzed using a scanning electron microscope (SEM). SEM evaluation of the tooth showed the three layers seamlessly; by increasing the enlargement the interface did not change.

Key words: adhesive, lithium disilicate, cementation, SEM.

Introduction

The use of lithium disilicate as ceramic material for fixed dental prosthesis combines good mechanical properties to excellent aesthetic results. This ceramic is indicated for single crowns, veneers and inlays, and compared with other glass-ceramics, it demonstrates very good performance (1).
Lithium disilicate represents a clinical option in both anterior and posterior region (2, 3), showing successful rates from 95.39 to 100% at 3 years (4).
It is generally assumed that clinical success of all-ceramic restorations is influenced by the type of cementation (5, 6). A strong point claimed for using lithium disilicate is adhesive cementation. As glass-ceramic, the inner surface of lithium disilicate may be etched by fluoridric acid to increase the surface energy and consequently the bond strenght (7, 8). After etching and cleaning the surface, a silane coupling agent is used to establish a chemical bond between luting composite and ceramic surface.
A recent study investigated the retentive strength of lithium disilicate crowns cemented on natural teeth (9). Posterior teeth restored with adhesively-cemented disilicate crowns most often failed by fracturing the root, suggesting a real adhesion between dental structure and ceramic reconstruction rather than merelya luting. Such an adhesive interface needs to be deeply investigated.
Aim of this work was to qualitatively evaluate the interface between tooth, luting composite and lithium disilicate surface using a scanning electron microscope (SEM).

Materials and methods

A restoration-free human molar, extracted for periodontal reason, was cleaned from dental plaque and periodontal tissues with ultrasonic instruments and curettes. The tooth was embedded in a self-curing acrylic resin block (ProBase Cold, Ivoclar Vivadent AG, Schaan, Liechtenstein) up to 2 mm below the cement-enamel junction (CEJ) and with its long axis perpendicular to the base of the block. The tooth was horizontally sectioned by using a diamond disk and 2 perpendicular grooves were made on the horizontal surface by using a spherical diamond bur, to facilitate the positioning of the restoration (Fig. 1).

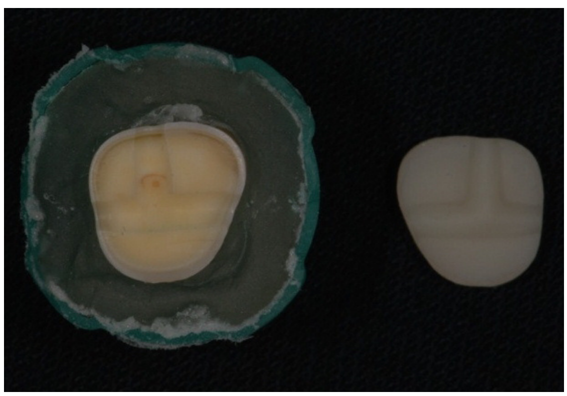

Figure 1. Preparation and restoration.

An anatomic onlay was waxed and hot pressed using lithium disilicate (IPS e.max PRESS LT A1, Ivoclar Vivadent AG, Schaan, Liechtenstein).
The inner surface of ceramic was etched with 5% hydrofluoridric acid (IPS Ceramic, Ivoclar Vivadent) for 20 second, then rinsed and cleaned in pure alcohol in ultrasonic bath for 10 minutes.
Therefore it was treated with universal primer (Monobond Plus, Ivoclar Vivadent AG, Schaan, Liechtenstein) and then dried with hot air.
The tooth was cleaned and dried, and then an adhesive (Multilink Primer, Ivoclar Vivadent AG, Schaan, Liechtenstein) was brushed on and dried. The cement (Multilink Automix, Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied on the inner surface of the ceramic and then the onlay was seated on the prepared teeth until the end of polymerization (Fig. 2)
After cementation the tooth was sectioned along the vertical axis using a diamond disk and the section was analyzed using a scanning electron microscope (SEM).

Figure 2. Cementation.

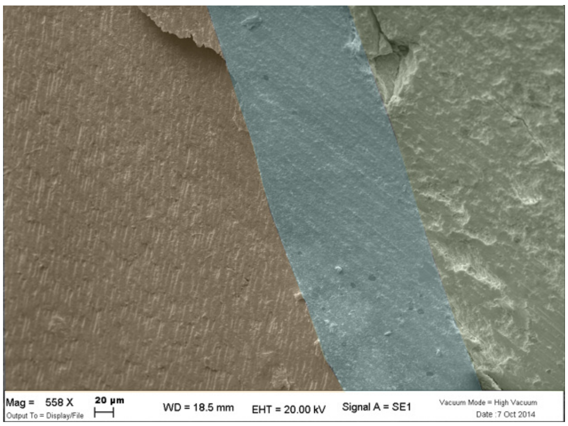

Figure 3. SEM evaluation at 558 X.

Results

SEM evaluation of the tooth section at 558 X (i.e. 20 µm) showed the three layers (tooth, luting composite and lithium disilicate) seamlessly (Fig. 3). With increasing enlargement at 800 X, 1.37 K X and until 3.67 K X, the interface did not change (Figs. 4-6).

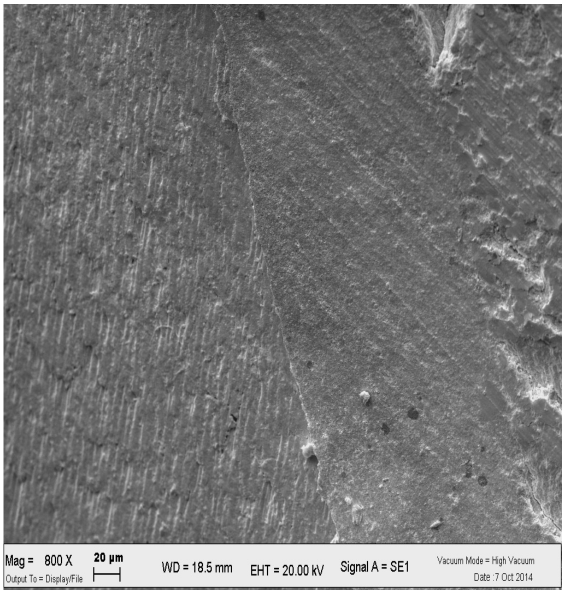

Figure 4. SEM evaluation at 800 X.

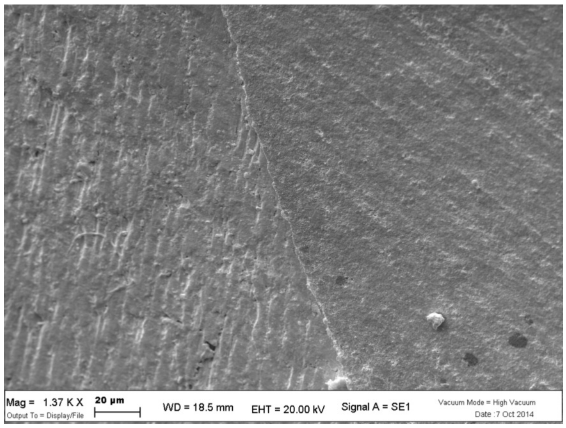

Figure 5. SEM evaluation at 1.37K X.

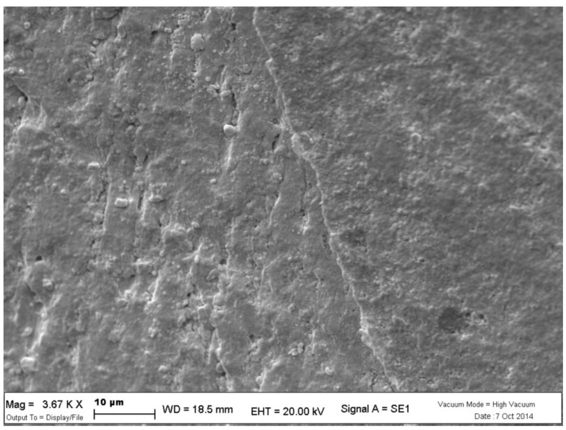

Figure 6. SEM evaluation at 3.67K X.

Discussion

Traditionally, definitive cementation is considered a fundamental step among restorative procedures. The correct cement selection and correct cementation procedure are crucial for adequate marginal sealing and proper retention of the restoration in time (10). In restoring teeth by using metal-free restoration, the cementation step acquires an additional value. In this case, indeed, cementation is not just a luting procedure, but it realises a true adhesion between two different materials, i.e. dental tissue and ceramic restoration. Using an adhesive cementation has a large impact on clinical decisions and procedures. First of all, it is possible to prepare a tooth with insufficient retention form: it happens very often in case of partial reconstructions (veneers or onlays), that would not be possible with traditional, not adhesive procedures. Furthermore, the adhesive interface between tooth and ceramic has the ability to increase the mechanical properties of the latter, improving the fracture resistance of the whole system (11). For this reason it is possible to use all-ceramic restorations also in load-bearing areas (e.g. posterior region).Therefore, it is recognized that the success of an all-ceramic restoration greatly depends on the quality of the adhesive interface. For this reason in the present study such an interface has been evaluated by SEM. The SEM analysis revealed a continuous interface from dentine to luting composite to lithium disilicate. Such evidence was confirmed by increasing the enlargement. Such a perfect interface may be most likely responsible of the very good performance of all-ceramic crowns in a recent study (9). Teeth restored with adhesively-cemented lithium disilicate crowns most often failed by fracture of the tooth instead of decementation of the crown in a *in vitro* pull-out test. Where adhesive cementation was not used, the most of the teeth failed by decementation.

Among glass ceramics, lithium disilicate has the highest flexural strength and for this reason has many clinical indications, also in high stress areas or conditions. Furthermore, disilicate as a glass ceramic may be chemical etched and this is a clear advantage relative to more performing polycrystalline ceramics as zirconia. For this reason a true adhesion to the tooth is possible to achieve with lithium disilicate and not with zirconia. A composite luting is mandatory to achieve adhesion both to the dental tissue and to inner ceramic layer (12, 13).

In conclusion, within the limits of this qualitative SEM evaluation, the adhesive interface between tooth, luting composite and ceramic appeared to be with no interruption. Further quantitative *in vitro* studies are needed to better address this issue.

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Original article

Fracture resistance of endodontically treated teeth restored with a bulkfill flowable material and a resin composite

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Summary

Aim. **To determine and compare the fracture resistance of endodontically treated teeth restored with a bulk fill flowable material (SDR) and a traditional resin composite.**
Methods. **Thirty maxillary and 30 mandibular first molars were selected based on similar dimensions. After cleaning, shaping and filling of the root canals and adhesive procedures, specimens were assigned to 3 subgroups for each tooth type (n=10): Group A: control group, including intact teeth; Group B: access cavities were restored with a traditional resin composite (EsthetX; Dentsply-Italy, Rome, Italy); Group C: access cavities were restored with a bulk fill flowable composite (SDR; Dentsply-Italy), except 1.5 mm layer of the occlusal surface that was restored with the same resin composite as Group B. The specimens were subjected to compressive force in a material static-testing machine until fracture occurred, the maximum fracture load of the specimens was measured (N) and the type of fracture was recorded as favorable or unfavorable. Data were statistically analyzed with one-way analysis of variance (ANOVA) and Bonferroni tests ($P<0.05$).**
Results. **No statistically significant differences were found among groups ($P<0.05$). Fracture resistance of endodontically treated teeth restored with a traditional resin composite and with a bulk fill flowable composite (SDR) was similar in both maxillary and mandibular molars and showed no significant decrease in fracture resistance compared to intact specimens.**
Conclusions. **No significant difference was observed in the mechanical fracture resistance of endodontically treated molars restored with traditional resin composite restorations compared to bulk fill flowable composite restorations.**

Key words: fracture resistance, endodontic treatment, bulk fill flowable composite, resin composite.

Introduction

The functional and aesthetic rehabilitation of endodontically treated teeth has been the subject of different studies (1). The restoration should not only provide function, aesthetic and marginal sealing, but also protect the remaining tooth structure (2, 3). Different studies have shown that the preparation of endodontic access cavities reduces the strength of the teeth, because of deep and extended cavity preparations which critically reduce the amount of dentin (4-8) and increase cuspal deflection during function (9). The importance of conserving the bulk of dentin was demonstrated in maintaining the structural integrity and in the prognosis of endodontically restored teeth (10-13), as the fracture resistance and stress distribution of endodontically treated teeth is directly affected by the amount of residual coronal dentin (4, 14-18).
In posterior preparations, especially when the cervical margin is located in dentin, the polymerization shrinkage effects can be significant, producing marginal defects and gaps despite careful application (19). Several techniques and a variety of restorative materials, which would minimize the stresses generated on the interface of the restoration by modifying some physical and mechanical properties have been proposed to reduce the effects of polymerization shrinkage (20-22). Furthermore, inadequate polymerization throughout the restoration may compromise its physical properties and increase elution of monomer

(23-26) and may lead to undesirable effects, such as gap formation, marginal leakage, recurrent caries. It may also negatively affect pulp tissue and may lead to premature failure of the restoration (27, 28).
Several manufacturers have recently developed and introduced new types of resin composites, so-called "bulk fill" materials, which can be applied to the cavity and light cured to a maximal increment thickness of 4 mm (29-32) with enhanced curing, shrinkage and physical properties (33). Bulk fill flowable resin composites are used in association with conventional composites for aesthetic restorations in posterior teeth, having lower polymerization stress, better flow with easy placement, an excellent adaptation to the cavity walls and low modulus of elasticity, which can reduce the stress generated on the cavity walls (34).
The purpose of this *in vitro* study was to compare the fracture resistance of endodontically treated upper and lower molars restored with direct traditional and bulk fill flowable resin composite restorations. The null hypothesis tested was that there was a difference in the fracture resistance and the mode of failure between endodontically treated maxillary and mandibular molars restored with traditional and bulk fill flowable resin composite.

Materials and methods

Sixty intact recently extracted human maxillary and mandibular molars with completely formed apices were used in this *in vitro* study. The exclusion criteria for tested teeth were the presence of caries, previous restoration and visible fracture lines or cracks. After a debridement with hand scaling instruments and cleansing with rubber cup and pomice, the teeth were stored in individually numbered containers with 0.1% thymol solution at 4° C until used. Thirty maxillary first molars with three separate roots and 30 mandibular first molars with two separate roots were selected based on similar anatomical crown height, measured from the occlusal surface to the cemento-enamel junction on the four sides of the teeth, and bucco-lingual (BL), mesio-distal (MD) dimensions at the occlusal surface. Tooth measurements were taken with a digital caliper. Preliminary radiographs were taken in two perpendicular directions (MD and BL) to determine root canal anatomy and measure the length and degree of canal curvature using the Schneider method (35). Specimens were subsequently assigned to 3 groups (n=10) for each tooth type creating homogenous groups considering the average of teeth dimensions in order to minimize the influence of size and shape variations on the results:

- Group A, the negative control group, which included 10 maxillary and 10 mandibular molars that were left intact for fracture testing, without any cavity preparation or root canal treatment;
- Group B, which included 10 maxillary and 10 mandibular molars, which were subjected to endodontic access cavity and endodontic procedures and were restored with a resin composite (EsthetX; Dentsply-Italy, Rome, Italy);
- Group C, which included 10 maxillary and 10 mandibular molars, which were subjected to endodontic access cavity and endodontic procedures and were restored with a bulk fill flowable composite (SDR; Dentsply-Italy), except 1.5 mm layer of the occlusal surface that was restored with the same resin composite as Group B.

The access cavity was prepared using water-cooled round-ended cylindrical diamond burs and non-end-cutting diamond burs mounted on a high-speed hand piece with different diameters. Root canals were negotiated with size 10 K-type files (Flexofile; Dentsply Maillefer, Ballaigues, Switzerland) to the major apical foramen and canals instrumented to length with NiTi rotary instruments (Mtwo; Sweden & Martina, Padova, Italy) up to the #25 tip size and 0.06 taper file. During the endodontic treatment 5.25% sodium hypochlorite (Niclor 5, Ogna, Muggiò Milan, Italy) for irrigation was intermittently deposited using Pro Rinse side-vented 30-G needles (Dentsply Tulsa Dental Specialties, Tulsa, OK). The canals were dried with paper points and filled with gutta-percha (single-cone #25/0.06 taper) and a resin-based endodontic sealer (AH-Plus, Dentsply Maillefer, Ballaigues, Switzerland). After the cleaning, shaping and filling procedures, post-operative radiographs were taken in the two perpendicular dimensions (MD and BL) to evaluate the endodontic treatment. Then, the enamel and dentin of the access cavity were etched with 37% phosphoric acid for 30 and 15 seconds respectively, rinsed for 30 seconds with a water/air spray, and gently air-dried to avoid desiccation. A light-polymerizing primer-bond adhesive (XP Bond, Dentsply International, York, USA) was applied, gently air-thinned and exposed to LED polymerization for 40 seconds. In group B access cavities were restored with direct resin composite (EsthetX; Dentsply-Italy, Rome, Italy) with material increments of maximum 2 mm. The specimens in Group C were restored with a bulk fill flowable composite with maximal increment thickness of 4 mm (SDR; Dentsply-Italy), except for 1.5 mm layer of the occlusal surface that was restored with the same resin composite as Group B (Fig. 1).
All the specimens were marked 2 mm below the cemento-enamel junction and were covered with approximately 0.25 mm-thick wax. The specimens were embedded in autopolymerizing acrylic resin (SR Ivolen; IvoclarVivadent, Schaan, Lichtenstein) in metallic cylindrical molds in position with their long axis parallel to that of the cylindrical molds. To simulate the periodontal ligament, at the first signs of the beginning of polymerization, the teeth were removed from the resin blocks and the wax was cleaned from the root surfaces. A standardized silicone layer was created using a light-body silicone-based impression material (Aquasil ultra light bodies, Dentsply International, York, USA) which was injected into the polymerizing resin bases. The teeth with now wax-free root surfaces were inserted into the resin bases immediately after the silicone injec-

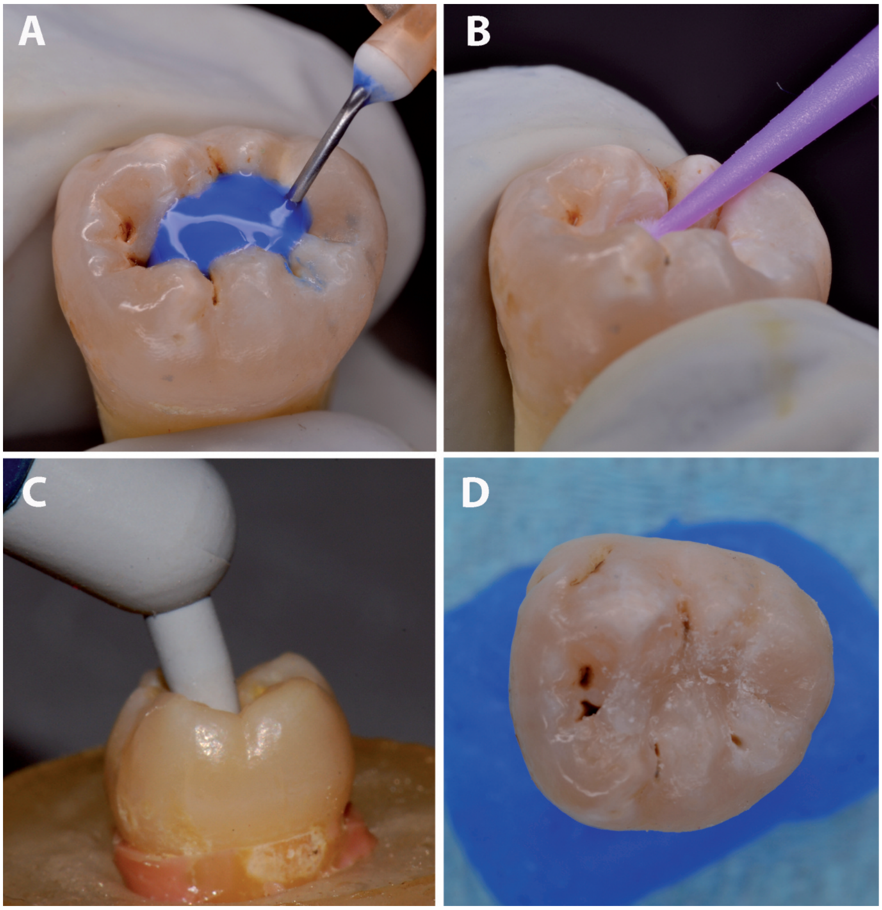

Figure 1. Representative images of enamel and dentin of access cavity etched with 37% phosphoric acid (A), application of a light-polymerizing primer-bond adhesive (B), application of a bulk fill flowable composite with maximal increment thickness of 4 mm (C) , and final restoration of 1.5 mm layer of the occlusal surface restored a traditional resin composite (D).

tion (36). All the specimens were stored in buffered saline plus 1.5% thymol at room temperature (24-28° C) until the fracture testing procedure.

All the 60 specimens were mounted in a mechanical material testing machine (LR30K; Lloyd Instruments Ltd, Fareham, UK) equipped by a (5k ± 5) N load cell. The teeth were loaded at their central fossa at a 30° angle to the long axis of the tooth (Fig. 2). The continuous compressive force at a cross- head speed of 1.6 mm/s was applied with a 6 mm diameter ball-ended steel compressive head until visible or audible evidence of fracture was shown. The force at fracture was measured in Newton (N) and type of fracture was recorded as "favorable" because restorable, when the failures were above the level of bone simulation (site of fracture above the acrylic resin) and as "unfavorable" because non-restorable, when the failures were extending below the level of bone simulation (site of fracture below the acrylic resin). The data were verified with the Kolmogorov-Smirnov test for the normality of the distribution and the Levene test for the homogeneity of variances. Thus, they were statistically evaluated by the analysis of variance test and Student-Newman-Keuls test for multiple comparisons (Prism 5.0; GraphPad Software, Inc, La Jolla, CA) with the significance level established at 5% ($P < .05$).

Results

The mean of the bucco-lingual (BL) and mesio-distal (MD) dimensions at the occlusal surface and the anatomical crown height of the teeth tested are presented in Table 1. No significant difference was found comparing all teeth dimensions in control and test groups ($P > .05$).

No statistically significant differences were found among groups ($P<0.05$). Fracture resistance of endodontically treated teeth restored with a traditional resin composite and with a bulk fill flowable composite (SDR) was similar in both maxillary (Group B: 1072±525N; Group C: 1241±388N) and mandibular molars (Group B: 1332±318N; Group C: 1527±449N). Restored teeth showed no significant decrease in fracture resistance compared to intact specimens similar in both maxillary (Group A: 1183±313N) and mandibular molars (Group A: 1620±170N) (Tab. 2).

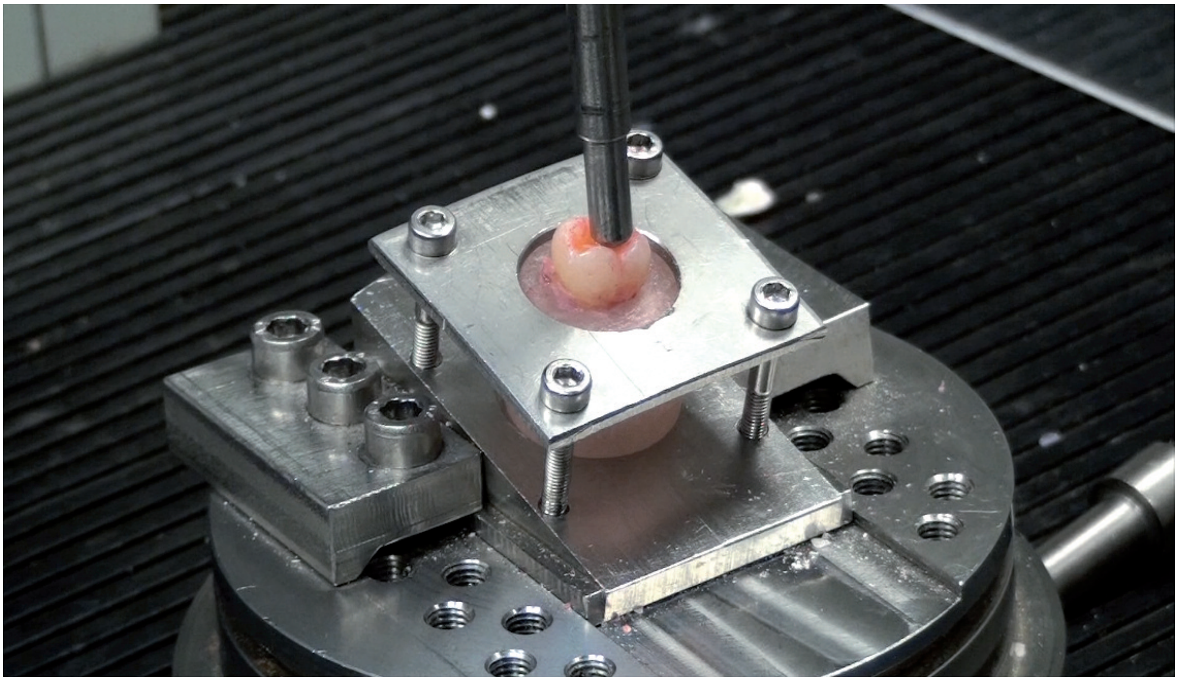

Figure 2. Simulated occlusal loading using a 6-mm-diameter steel sphere placed on the central fossa with lingual orientation in axio-occlusal line at 30° angle to the long axis of a mandibular molar tooth.

Table 1. Mean and Standard Deviation of the Mesio-Distal (MD) and Buccal-Lingual (BL) dimensions and the anatomical crown height (measured at the four sides of the tooth) of the tested teeth in each group.

Groups	**Control**			**Bulk fill material (SDR)**			**Traditional resin composite**		
Tooth Type (n=10)	Occlusal Surface		Anatomical Crown Height	Occlusal Surface		Anatomical Crown Height	Occlusal Surface		Anatomical Crown Height
	MD	BL		MD	BL		MD	BL	
Upper Molars	9.9^{a} (0.6)	9.7^{a} (0.7)	5.4 $(0.1)^{b}$	10.0^{a} (0.5)	10.1^{a} (1.1)	5.6 $(0.6)^{b}$	9.9^{a} (0.5)	10.1^{a} (0.9)	5.4 $(0.4)^{b}$
Lower Molars	10.7^{a} (1.2)	10.3^{a} (0.6)	5.7 $(0.4)^{b}$	10.6^{a} (0.8)	10.1^{a} (0.7)	5.5 $(0.4)^{b}$	10.4^{a} (1.2)	10.2^{a} (0.8)	5.6 $(0.7)^{b}$

Similar upper letter case in the same row indicates no statistically significant differences (P > .05).

Table 2. Load at fracture (mean ± standard deviation) and type of fracture, Favorable (F) or Unfavorable (U) for intact teeth (control, Group A), and teeth restored with traditional resin composite (Group B) or with bulk fill flowable material (SDR) (Group C) assessed after the static test using the Instron Universal Machine.

Tooth Type (n=10)	**Load at Fracture (N)**			**Type of Fracture**					
				F	**U**	**F**	**U**	**F**	**U**
	Group A	Group B	Group C	Group A		Group B		Group C	
Upper Molars	1172 $(598)^{a}$	1001 $(453)^{a}$	1313 $(428)^{a}$	8^{a}	2^{b}	3^{b}	7^{a}	3^{b}	7^{a}
Lower Molars	1572 $(639)^{a}$	1375 $(310)^{a}$	1484 $(471)^{a}$	7^{a}	3^{b}	3^{b}	7^{a}	2^{b}	8^{a}

Similar upper letter case in the same row indicates no statistically significant differences (P > .05).

60.8% of the failures in total were unfavorable (Group B 70%; Group C, 75%; control group, 25%). No significant differences were found in the mode of failure of the differently restored teeth between Group B and Group C, while intact teeth presented significantly more favorable fractures compared to restored specimens (both Group B and Group C).

Discussion

The null hypothesis investigated in the present study can be rejected, as the results obtained support that there is no difference in the fracture resistance and in the mode of failure between endodontically treated maxillary and mandibular molars restored with a bulk fill flowable resin composite (SDR) or a traditional resin composite.

Fracture susceptibility of root-filled teeth is affected mostly by the amount of the remaining dentin (4, 37) and it is not related to its biomechanical properties after endodontic treatment, such as hardness and toughness (38). Some studies have shown that the reduction of tooth structure results in weaker teeth due to restorative procedures (6-8). However, according to Reeh et al. (4), endodontic procedures have only a small effect on the tooth, reducing the relative rigidity by 5%, which is contributed entirely by the access opening. Restorative procedures and, particularly, the loss of marginal ridge integrity, were the greatest contributors to loss of tooth resistance. The loss of 1 marginal ridge resulted in a 46% loss in tooth rigidity, and a MOD preparation resulted in an average loss of 63% in relative cuspal rigidity.

Several studies were conducted to determine the ideal materials and techniques to restore endodontically treated teeth because their long-term prognosis depends on the quality of the final restoration (39-42). Usually, to restore endodontically treated teeth several resin increments are required to fill the cavity preparation because of the large volume of the restoration. Thus, the clinician must compensate the polymerization shrinkage of traditional resin-based composite, by filling the cavities in several increments (43). A new category of flowable resin-based composites has been introduced as bulk fill base material that can be applied in 4 mm thick bulks instead of using the incremental placement technique, without negatively affecting the polymerization shrinkage, cavity adaptation or the degree of conversion (30).

The results of the present study show that there were no significant differences in the static fracture resistance of endodontically treated molars restored with bulk fill flowable resin composite (SDR) and a traditional resin composite. Moreover, the mean fracture load for teeth restored with SDR was higher compared with the mean fracture load of specimens restored with traditional resin composites, without any statistical significance. Furthermore, the results of this study showed that there was no significant difference between teeth restored with SDR and intact teeth. These findings may be attributed to the elastic buffer effect of using low viscosity flowable composite and the characteristic low contraction stress and low modulus of elasticity of SDR flow (44, 45). High flexural modulus can inhibit the ability of a material to resist deformation due to loading and promote the accumulation of surface and bulk defects, which may lead to premature failure (46, 47). These findings are in agreement with those of Atiyah et al. (48), who reported increased fracture resistance of endodontically treated premolars restored with SDR.

In the present study 75% of the samples in the intact control teeth presented favorable fracture type that was an important statistical difference with the restored groups. In fact, the majority of the teeth restored with SDR (75%) and with a traditional resin composite (70%) reported unfavorable type of fracture. However, no significant differences were found in the mode of failure between restored teeth. Furthermore, all failures of the restored teeth were cohesive fractures, regardless of the type of restoration. The low elastic modulus may explain the severity of fracture type presented in restored teeth groups and the occurrence of unfavorable fracture. These findings are in agreement with previous reports that found an increased frequency and severity of cuspal fracture due to removal of cervical dentin (49).

The limitations of this study must be recognized. The experimental methods used for *in vitro* analyses do not accurately reflect intraoral conditions, in which failures occur primarily due to fatigue. Future research in this area should use cyclic loading and other fatiguing simulation to more accurately reproduce the clinical environment. Additional clinical studies are necessary to determine the long-term prognosis of endodontically treated maxillary and mandibular molars restored with bulk fill flowable resin composite.

Within the limitations of this *in vitro* study, endodontically treated upper and lower molars restored with bulk fill flowable resin composite presented a resistance to fracture under simulated compressive force not significantly different than that of traditional resin composite restorations. Restored teeth showed no significant decrease in fracture resistance compared to intact specimens. Furthermore, no differences were found in the mode of failure of the differently restored teeth, while intact teeth presented statistically more favorable fractures.

Bulk fill flowable composites can be used to restore endodontically treated posterior teeth using 4 mm maximum increments and 1.5 mm occlusal traditional layer because this does not reduce the mechanical resistance of the restored teeth, while making the procedure easier, less stressful and with a reduced chair side time.

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The Authors deny any conflicts of interest.
The Authors affirm that we have no financial affiliation

(e.g., employment, direct payment, stock holdings, retainers, consultant ships, patent licensing arrangements or honoraria), or involvement with any commercial organization with direct financial interest in the subject or materials discussed in this manuscript, nor have any such arrangements existed in the past three years. Any other potential conflict of interest is disclosed.

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