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Effects of Oxalate Desensitizer with Different Resin Cement-Retained Indirect Composite Inlays on Fracture Resistance of Teeth

Fereshteh Shafiei, DMD, MSc,[1] Ali Asghar Alavi, DMD, MSc,[2] Fatemeh Karimi, DMD, MSc,[3] & Elham Ansarifard, DMD[4]

[1]Assosciate Professor, Department of Operative Dentistry, School of Dentistry, Shiraz University of Medical Sciences, Iran
[2]Professor, Biomaterials Research Center, Department of Operative Dentistry, School of Dentistry, Shiraz University of Medical Sciences, Iran
[3]Postgraduate Student, Department of Operative Dentistry, School of Dentistry, Shiraz University of Medical Sciences, Iran
[4]Postgraduate Student, Department of Prosthodontics, School of Dentistry, Shiraz University of Medical Sciences, Iran

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Correspondence
Fatemeh Karimi, School of Dental Medicine, Department of Operative Dentistry, Shiraz University of Medical Sciences, Shiraz, 7186783694, Iran. E-mail: karimifatemeh14@yahoo.com

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Abstract

Purpose: This study investigated whether the tubular occluding effect of oxalate desensitizer (OX) during adhesive cementation (three resin cements) influenced fracture resistance of teeth restored with adhesive inlays.
Materials and Methods: Ninety intact maxillary premolars were randomly divided into 9 groups of 10 each. The two control groups were Gr 1, intact teeth and Gr 2, mesio-occlusodistal preparation only. In six experimental groups, the composite inlays were cemented with ED Primer II/Panavia F 2.0, Excite DSC/Variolink II, and One-Step Plus/Duolink according to manufacturers' instructions (Groups 3, 5, and 7, respectively) or with OX during cementation (Groups 4, 6, and 8, respectively). In Group 9, inlays were cemented with a resin cement without adhesive system. After thermocycling, fracture strength was tested. The data were analyzed using two-way and one-way ANOVA and LSD post hoc tests ($\alpha = 0.05$).
Results: Fracture resistance of the six groups were significantly affected by OX ($p = 0.002$) but not by the resin cement type ($p > 0.05$). The interaction of the two factors was statistically significant ($p = 0.052$). A statistically significant difference between all groups was found ($p < 0.001$). The mean fracture resistances (N) were: Gr1 = 1168 ± 157,[a] Gr2 = 360 ± 110,[d] Gr3 = 1026 ± 188,[b] Gr4 = 887 ± 143,[c] Gr5 = 1007 ± 132,[b] Gr6 = 810 ± 164,[c] Gr7 = 1033 ± 218,[a] Gr8 = 955 ± 147,[ab] Gr9 = 780 ± 86[c] (groups with the same superscript letter indicate statistical similarity).
Conclusions: Combining an OX with three resin cements had a significant negative effect on the fracture resistance of premolars restored with composite inlay cemented with Panavia F2.0 and Variolink II, but it had no significant effect when cemented with Duolink.

Removal of tooth structure during cavity preparation can lead to a decrease in fracture resistance of the prepared tooth. When the continuity of the enamel is broken, the risk for tooth fracture is increased. Hence, the restorative material should recreate the original resistance after restoration.[1-3]

Considering patients' increasing demands for esthetic restorations, resin composites with bonding ability have been widely used in clinical practice; however, the major shortcoming of resin composite is high polymerization shrinkage stress, resulting in marginal gaps and microleakage, especially when the gingival margin is located in the dentin.[4] Moreover, this shrinkage can lower the fracture resistance of weakened cusps.[5] To overcome these problems and technical difficulties associated with the placement of extensive restorations, indirectly placed composite inlays have been proposed.[4,6] These restorations are bonded to the tooth structure by adhesive resin cements.[1-2] Effective bonding of resin cements to restoration materials and the tooth structure are necessary to guarantee a successful restoration. This bonding reinforces substrates, increases retention,[7] and decreases microleakage, resulting in reduced postoperative sensitivity.[8] Although composite inlay/onlay technique is considered a suitable treatment for restoring medium/large cavities,[6,9] the literature indicates insignificant differences in clinical performance and postoperative sensitivity between these restorations and direct composite restorations.[6,9-14] A trend to more postoperative sensitivity was reported for inlay teeth compared with teeth restored with direct composite.[13] The most important postrestorative that poses a frequent problem in practice is hypersensitivity after luting of adhesive indirect restorations.[8]

Reduction of the vital tooth structure during preparation for indirect restorations exposes a large number of tubules. Etch-and-rinse and self-etch adhesive systems, used to bond the resin cement to the tooth structure,[15] lead to initial sealing of the exposed tubules. Simplified versions of these adhesives have been marketed to provide simpler and faster adhesion procedures.[16] However, the outward flow of dentinal fluid from the tubules can interfere with resin monomer infiltration, especially with etch-and-rinse adhesives that remove the smear layer.[17] Air drying the adhesive and the use of solvated ionic comonomer mixtures of the adhesive induce outward evaporative and osmotic fluid transudation, respectively, before polymerization of the adhesive.[16,17] This transudation through the hybrid and adhesive layers may entrap water blisters along the adhesive/resin interface.[18] During mastication, the resultant rapid fluid flow can cause postoperative sensitivity.[19] Furthermore, after polymerization of the adhesive and during slow setting of dual-cure resin cements, water can diffuse from the underlying hydrated dentin across the simplified adhesives via an osmotic gradient.[16] This water may interfere with resin cement polymerization by inducing emulsion polymerization of the hydrophobic luting resin cement.[16,18] This phenomenon is one of two main factors responsible for the incompatibility between auto/dual-cured resin cements and simplified adhesives.[16] As a result of water permeation, bonded indirect restorations may become partially decoupled.[18] Imperfection of the bond might also influence the resistance to fracture.

Dentin hypersensitivity is reduced when the dentinal tubules are intrinsically blocked by the precipitation of water-insoluble materials.[20] Fluoride and/or HEMA-containing desensitizing agents, Gluma desensitizer, varnish, resin-containing oxalic acid, and resin-free oxalate desensitizing agents have been reported to be used along with resin cements.[20–22] However, some components in desensitizer agents may induce chemical interactions with the dentin, impairing the subsequent interactions between the dentin and resin cement.[20] This interference may decrease the bond strength of resin cements to desensitizer-treated dentin.[20–22] Therefore, one way suggested to relieve postoperative sensitivity is subsurface tubular occlusion with the application of occluding agents that do not interfere with subsequent resin infiltration or result in the formation of hybridized resin tags.

Oxalate desensitizer (OX) has been marketed as an acidic resin-free oxalate potassium solution or gel. When OX is applied to acid-etched dentin, calcium oxalate crystals occlude the dentin tubules 5 μm to 10 μm below the surface, and the dentin surface is available for bonding with adhesives. Therefore, OX does not compromise the bond strength of relatively neutral etch-and-rinse adhesives.[23,24] The potential advantage of tubular occlusion during bonding with adhesive resin cements is that it limits interference with resin infiltration by tubular fluid flow and facilitates solvent and water evaporation.[24] Thus, OX may enhance the formation of a homogenous hybrid layer throughout the whole depth of the demineralized wet dentin. Nevertheless, different effects of OX application on the sealing/bonding efficacy of adhesive systems[24–28] or resin cements with various compositions and pH have been demonstrated.[21,29]

Adhesive restorations should not only provide a marginal seal, but should also increase strength of the weakened structure.[30–32] To provide these two requirements, stabilizing the effective and defect-free adhesion between the restoration and cavity wall is of major importance.[32]

The loss of approximately 60% or 50% of tooth resistance was demonstrated following MOD cavity preparation.[33–35] It is noteworthy that when prepared teeth are restored with adhesive material and cemented by adhesive resin cement, a partial or total recovery of fracture resistance can be obtained.[33–36] Levels of resistance recovery have been reported to be between 4% and 97%, depending on direct[32,37–39] or indirect restorative material, cement type, and experimental design.[31,33–36,40,41] In the literature, fracture resistance of the adhesive-restored premolar versus the intact premolar has been reported as follows: 384 ± 141 versus 844 ± 328 N,[33] 1810 ± 500 versus 1910 ± 200 N,[34] 1033 ± 307 versus 1312 ± 210 N,[35] and 1424 ± 550 versus 1176 ± 199 N.[30] The formation of a unique body between restoration and tooth structure is the main factor for the higher resistance.[31] This integrity depends on achieving the high bonding capacity between resin cement with tooth structure and with composite.[31,34,42] Furthermore, the high capacity of composite to absorb and distribute loading forces in a homogenous way is an important factor.[34] Therefore, any imperfection of these bonds may lead to a decreased resistance to fracture of a restored unit.

To date, no studies have investigated the effect of the combination of OX with adhesive cements on fracture resistance of restored teeth by adhesive composite inlays. We therefore designed this study to evaluate whether the tubular occluding effect of OX during adhesive cementation (a self-etch cement, Panavia F2.0, and two etch-and-rinse cements, Variolink II and Duolink) influenced fracture resistance of teeth restored with adhesive inlays. In this evaluation, maxillary premolars were used, since among the posterior teeth, these teeth are positioned in the esthetic zone in the dental arch so that they need a tooth-colored restoration,[3] and their anatomic shape facilitates separation of the cusps under occlusal loading.[30] The null hypothesis was that combining OX with adhesive resin cements during cementation would have no effect on the fracture resistance of restored teeth with inlays cemented with different cements.

Materials and methods

Following approval of the research protocol by the local ethics committee, 90 sound, noncarious, single-root maxillary premolars extracted for orthodontic treatment were used. The roots and crowns of the teeth were similar in size (buccolingual, mesiodistal and gingivo-occlusal dimensions) and were stored in 0.5% thymol solution at 4°C. The cleaned teeth were carefully inspected under a stereomicroscope (Carl Ziess, Oberkochen, Germany) at 20× magnification to exclude teeth with defects, such as fracture lines. The teeth were then randomly divided into nine groups of 10 teeth each and subjected to the following procedures:

Group 1: Unaltered intact teeth were used as the negative control (G1, NC).

Table 1 Resin cement systems used and their application procedure

Resin cement (batch#; manufacturer)	Adhesive system (batch#; category)	Dentin pretreatment	Composite pretreatment	Luting agent mixing
Panavia F2.0 (Paste A/00447A, Paste B /00080A; Kuraray Inc, Tokyo, Japan)	ED primer II (A/00286A, B/00160B; one-step self-etch)	Mix one drop of each ED primer liquid A and B for 5 sec, air dry gently after 60 sec	Etch for 5 sec, rinse, air dry, mix one drop of each Clearfil SE primer and Porcelain Bond Activator for 5 sec, apply.	Mix universal and catalyst paste for 20 sec, light cure for 20 sec, after removing excess cement, apply Oxyguard for 3 min
Variolink II (K10496; Ivoclar Vivadent, Schaan, Liechtenstein)	Excite DSC (M52582; two-step etch-and-rinse)	Etch for 15 sec, rinse, gently air dry, apply the adhesive for 10 sec, air dry and light cure for 20 sec.	Etch for 15 sec, rinse, air dry, apply Monobond S for 60 sec, air dry.	Mix base and catalyst paste for 10–20 sec, light cure for 20 sec.
Duolink (09000011311/; Bisco, Schaumburg, IL)	One-Step Plus (0800004236; two-step etch-and-rinse)	Etch for 15 sec, rinse, gently air dry. Shake the bottle for 3–5 sec. Apply two consecutive coats, agitate 20 sec, gently air dry, light cure for 10 sec	Etch for 15 sec, rinse, air dry, apply Monobond S for 60 sec, air dry.	Mix base and catalyst paste for 10-20 sec, light cure for 20 sec.

In groups 2 to 9, mesio-occlusodistal cavities were prepared with the gingival margin located 1 mm coronal to the cementoenamel junction (CEJ), using a diamond bur (#7875, Teeskavan, Iran) in a high-speed handpiece under air-water spray, producing divergent cavity walls. After every five preparations, the diamond burs were replaced. The buccolingual width of each cavity at the isthmus was half the intercuspal distance, and its depth was 2 mm at the distal and mesial pits. Each box had a gingival floor with axial depth of 1.5 mm and a 2 mm high axial wall. Measurements were made with a digital caliper (Mitutoyo Digimatic, Mitutoyo, Kawasaki, Japan) at 0.1 mm sensitivity for proper and accurate standardization of cavity dimensions.

Group 2: MOD-prepared-only. These teeth were not restored and were used as the positive control (G2, PC).

In groups 3 to 9, an impression of the crown was taken with a poly(vinyl siloxane) (PVS) impression material (Speedex, ColtèneWhaledent AG, Attstatten, Switzerland) to produce a hard stone working model. The inlays were fabricated with a composite resin (Z250, 3M ESPE, St. Paul, MN) using the oblique incremental technique. The curing was done with a halogen light unit (Coltolux, ColtèneWhaledent) at a 500 mW/cm^2 intensity. Light intensity output was checked every ten restorations with a radiometer from the same manufacturer. The composite inlays were then removed from the model after initial curing and further polymerized in an oven. The internal surfaces of the inlays were sandblasted with 50 μm alumina particles (Dento-Prep, Ronvig, Denmark), washed, and air-dried. Then a silane-coupling agent was applied. All the preparations and restorations were performed by the same operator. Throughout the experiment, teeth were handled in moist gauze and stored in an incubator at 37°C and 100% humidity to prevent dehydration.

Each tooth was embedded in a cylinder of self-curing acrylic resin (Acropars, Tehran, Iran) surrounded by PVS impression material up to 1 mm apical to the CEJ, with the long axis of the tooth perpendicular to the base of the cylinder.

Groups 3, 5, and 7: The inlays were cemented with ED Primer II/Panavia F 2.0, Excite DSC/Variolink II, and One-Step Plus/Duolink, respectively, according to manufacturer's instructions (Table 1).

Group 4: OX (BisBlock, Bisco, Schaumburg, IL, Batch #1000000152) was first rubbed onto the dentin surfaces for 30 seconds. After rinsing and drying, ED Primer II was applied, and the inlays were cemented with Panavia F 2.0 similar to group 3. The main component of commercial OX products is 3% oxalic acid as a gel or solution. The other component is potassium salt. Reviewing the literature, BisBlock gel and SuperSeal liquid were used most often, as they were more available.[24–29] The gel product used is more easily applicable than the liquid one.

Groups 6 and 8: After acid etching, OX was applied. Then, the teeth were rinsed and gently dried, and Excite DSC/Variolink II and One-Step Plus/Duolink were used, respectively, for cementation of the inlays.

Group 9: After acid etching, rinsing, and drying, the inlays were cemented with Duolink cement without adhesive system.

After 24 hours of storage for complete polymerization, the specimens were thermocycled for 1000 cycles at 5°C and 55°C with a 30-second dwell time according to ISO TR 11454 (1994). This minimal thermocycling regimen was done because the effect of association of OX with different adhesive cements on the initial fracture resistance was compared. A static fracture resistance test was performed using a universal testing machine (Zwick-Roell, Zwick, Ulm, Germany). The specimens were subjected to a continuous compressive force at a 1 mm/min crosshead speed. The force was applied by a 4.8-mm-diameter round metal bar positioned parallel to the long axes of the teeth, in contact with the occlusal slopes of the cusps without touching

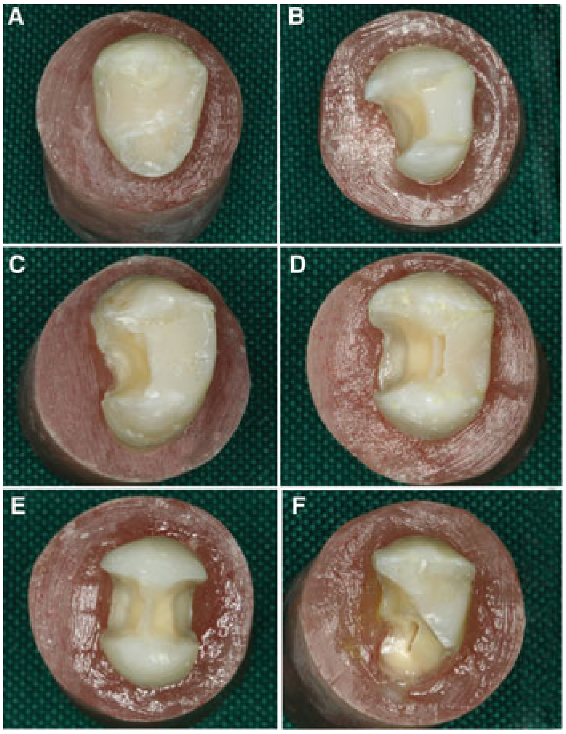

Figure 1 (A) Fracture mode I. (B) Fracture mode II. (C) Fracture mode III. (D) Fracture mode IV. (E) Fracture mode V (fracture of inlay). (F) Fracture mode V (fracture of tooth and inlay).

Table 2 Fracture resistance (mean ± SD)

Group	Group code	Mean (SD) (N)
1	NC (Intact teeth)	1168 (157)[a]
2	PC (Prepared teeth)	360 (110)[d]
3	Panavia F 2.0 (without OX)	1026 (188)[b]
4	Panavia F 2.0 (with OX)	887 (143)[c]
5	Variolink (without OX)	1007 (132)[b]
6	Variolink (with OX)	810 (164)[c]
7	Duolink (without OX)	1033 (218)[a]
8	Duolink (with OX)	955 (147)[ab]
9	Duolink (without adhesive)	780 (86)[c]

Groups with the same letter were not significantly different ($p > 0.05$).

Table 3 Mode of fracture in the seven restored groups

Group		N	I	II	III	IV	V
3	Panavia F 2.0 (without OX)	10	0	1	4	3	2
4	Panavia F 2.0 (with OX)	10	1	1	7	1	0
5	Variolink (without OX)	10	1	0	4	2	3
6	Variolink (with OX)	10	1	2	6	1	0
7	Duolink (without OX)	10	0	0	2	3	5
8	Duolink (with OX)	10	0	2	2	3	3
9	Duolink (without adhesive)	10	1	3	6	0	0

Mode I Minimal fracture of tooth or inlay; Mode II Fracture less than half of inlay; Mode III Fracture through midline of inlay; Mode IV Fracture more than half of inlay; Mode V Severe fracture of tooth and/or inlay.

the inlays. Peak load to fracture for each tooth was recorded in Newtons. The data obtained from groups 3 to 8 were analyzed with two-way ANOVA for the effect of resin cement and OX. Since groups 1, 2, and 9 cannot be included in the framework of this test, one-way ANOVA and post hoc LSD tests were done to compare the differences among all groups at a significance level of 0.05. All statistical analyses were done with SPSS Version 11.5 software (IBM, Armonk, NY).

The fractured specimens in restored groups were then examined by two independent operators to determine the mode of fracture, as described by Burke et al.[2] Mode I: Minimal fracture of tooth or inlay; Mode II: Fracture less than half of inlay; Mode III: Fracture through midline of inlay; Mode IV: Fracture more than half of inlay; Mode V: Severe fracture of tooth and/or inlay (Fig 1).

Results

Fracture resistance (mean ± SD) for the nine groups are shown in Table 2. The results of two-way ANOVA revealed that the fracture resistance results of the six groups were significantly affected by OX ($p = 0.002$) but not by the resin cement type ($p > 0.05$). The interaction of the two factors was statistically significant ($p = 0.052$). Comparisons with one-way ANOVA revealed significant differences in fracture resistance among the tested groups ($p < 0.001$). The fracture mode of the seven restored groups is shown in Table 3. More severe fractures (Mode IV and V) were observed in the groups with higher fracture resistance values (groups 3, 5, 7, 8).

Discussion

In the current study, the weakening effect of cavity preparation and strengthening effect of adhesive restorations were confirmed so that a nonrestored MOD cavity significantly decreased the fracture resistance (360 ± 110 N vs. 1168 ± 157 N), while adhesive inlay restoration partially/totally recovered resistance to fracture (1033 ± 218, 1026 ± 188, 1007 ± 132 N for three cements). The inlay cemented with Duolink showed a strength approximately similar to that of the intact teeth; however, the inlay cemented with Panavia and Variolink showed lower strength than that of the intact teeth. These values were approximately comparable to those reported by Santos and Bezerra.[35]

On the other hand, inlay cementing without an adhesive system associated with the resin cement could lead to a decrease in the fracture resistance compared to adhesive application. These results indicated a substantial role of the adhesive joint between the cement and tooth structure, via formation of a hybrid layer, in the effectiveness of luting cement for strengthening of the teeth restored with adhesive inlay.[42]

In the present study, OX pretreatment during inlay cementation resulted in decreased fracture resistance for three adhesive cements. This decrease was statistically significant for Panavia F2.0 and Variolink, but not for Duolink (in pairwise comparisons employed following ANOVA analysis of the nine groups, however, the interaction of OX and cement showed a borderline *p* value, 0.052). On the basis of previously mentioned points, this difference can be related to the interfering effect of OX on bonding ability of adhesive systems associated with Variolink and Panavia F2.0 to dentin. The low pH of Ex DSC may have dissolved the calcium oxalate crystals formed in the dentinal tubules, compromising the formation of hybridized resin tags and the hybrid layer. Moreover, the ethanol content as a solvent in this adhesive may increase the solubility of calcium oxalate in an ethanol-water mixture.[43] This adverse effect on the marginal leakage of the composite restoration bonded with low-pH Excite was reported in a previous study.[25]

Ex DSC is a dual-cure version of Excite with similar components, except that Ex DSC has an additional initiator coated on the brush supplied in the package. In another study,[32] the marginal leakage was demonstrated as an indication of a defective adhesive interface, thereby an indication of weakening of the restored tooth. Imperfections of the bond correlated with reduction in fracture resistance. Earlier work found that the tubular blocking capacity of OX treatment on acid-etched dentin deteriorated after the low-pH fluoride-containing adhesives were applied,[44] and that this reduced their bond strength.[26] This incompatibility may be related to the formation of spherical globules (CaF_2 material) following interaction of the free fluoride ions present in the adhesives with calcium and phosphate ions on the dentin surface. The low pH of the adhesive is critical for this interaction because the dentin surface is completely depleted of calcium phosphate after acid etching. The calcium ions may have become available from the dissolution of calcium oxalate crystals by the low-pH adhesives.[26,44] The OS Plus adhesive, which is less acidic (pH = 4.61), contains fluoride (806 ppm).[25] In the current study, OX pretreatment associated with OS Plus/Duolink did not significantly alter the fracture resistance of inlay cemented with Duolink cement. This finding was consistent with the manufacturer's instructions, which recommend the application of BisBlock in combination with One-Step or One-Step Plus. One recent study reported that OX, when applied with OS Plus, had no negative effect on the sealing ability of composite restorations.[25] Two dentin permeability studies[23,44] and three bond strength studies[23,24,26] found that One-Step (which contains no fluoride) was compatible with two or three resin-free oxalates. In a recent study, the compatibility of One-Step Plus/Variolink II with OX resulted in a beneficial occluding effect of OX. This effect reduced the adverse effect of tubular fluid flow on dentin bond strength when chemical curing or delayed light curing of the resin cement were used.[29]

In contrast to these results, despite the relatively high pH values and lack of fluoride content in the two adhesives used, an adverse effect of OX on the initial bond strength of Single Bond and One-Step and long-term bond strength of Single-Bond was reported.[28-45] In a recent study, this effect was observed with Single Bond, but with low-pH Prime and Bond NT, OX had no effect on its bonding.[27] These results were attributed to other factors, including the additional etching effect of oxalic acid on dentin and inhibiting effect of remaining oxalic acid on adhesive polymerization regardless of the adhesive type,[28] solubility of calcium oxalate after 3 months of simulated pulpal pressure,[45] and curing characteristic and solvent type of the adhesive.[27]

On the other hand, the application of low-pH OX (1.5–1.8) on smear-layer-covered dentin followed by rinsing resulted in removal of the smear layer and replacement with an acid-resistant calcium oxalate crystals layer.[21,23] Scanning electron microscopic (SEM) observations showed blocking of some dentinal tubules—approximately half of the tubules appeared to be closed after OX application.[21,46] These alterations in dentinal surface were considered to have an adverse effect on bonding of ED Primer/Panavia F2.0 to dentin, thereby decreasing the fracture resistance; however, the lower bond strength of Panavia F2.0 compared to the control group after OX solution application reported by Huh et al was not significant.[21]

The limitations of the experimental design of the present study must be recognized. This design may not accurately simulate intraoral conditions. Although the teeth were simply hydrated, no pulpal pressure was involved. Furthermore, the loading used in this fracture test did not reproduce typical mastication forces, as a continually increasing axial force was applied until the fracture occurred. This compressive static loading is different from the dynamic fatigue loading typical of mastication; however, this test can be an important source of information about structural integrity. The methodology used in our study was designed in a macroenvironment, which cannot detect any interaction that actually occurs between OX and adhesive system on dentine surfaces. This method can be an indirect indicator of the effect of OX application on the quality of the bonding to tooth structure during adhesive cementation of inlay restorations. The actual interfaces created with OX might be examined using SEM analysis. Considering this effect on the clinical performance of adhesive inlay, further investigations are recommended to be performed over the long term with simulation of thermal and mechanical fatigue before OX pretreatment during inlay cementation with resin cements.

Conclusions

Within the limitations of the present study, the following could be concluded:

(1) According to the results, the tested hypothesis was partially rejected.
(2) Among the three adhesive resin cements examined, only OS Plus/Duolink combined with OX pretreatment exhibited fracture resistance approximately similar to the control group. This strength reached that of the intact teeth.

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