ORIGINAL RESEARCH

Influence of Cavity Pretreatments on the Fracture Resistance of Premolars with Self-adhesive Cemented Composite Inlay

Fereshteh Shafiei[1], Zahra Jowkar[2], Neda Hosseini[3]

Abstract

Aim: The aim of this study is to investigate whether different cavity pretreatment approaches affect the strength of premolars restored with self-adhesive (SA) resin cemented-composite resin inlays after mechanical and water aging.

Materials and methods: A total of 120 intact maxillary premolars were divided into 10 groups ($n = 12$). Mesio-occluso-distal (MOD) cavities were prepared in the teeth of nine groups, except group I in which the teeth remained intact. In group II, cavities were unrestored. Following fabrication of composite resin inlays for groups III–X, in group III, the inlays were cemented using the etch-and-rinse (E and R) adhesive/conventional resin cement. In other groups, cementation was performed using a SA cement with or without cavity pretreatments as follows: group IV: SA cement alone, group V: acid etching of enamel and dentin, group VI: acid etching of enamel, group VII: universal adhesive in the selective enamel-etching mode, group VIII: universal adhesive in the E and R mode, group IX: ethylenediaminetetraacetic acid (EDTA) conditioning, and group X: 20% polyacrylic acid conditioning. After aging processes, static fracture resistance was tested. Data were analyzed using one-way ANOVA and Dunn tests ($\alpha = 0.05$).

Results: Fracture resistance of the 10 groups yielded a significant difference ($p < 0.001$). The median fracture resistances in Newton were the following: Gr I = 1025^{A}, Gr II = 311^{BC}, Gr III = 785^{A}, Gr IV = 500^{B}, Gr V = 435^{B}, Gr VI = 775^{A}, Gr VII = 805^{A}, Gr VIII = 411^{BC}, Gr IX = 397^{BC}, and Gr X = 312^{C}.

Conclusion: Unlike the conventional method, SA cementation could not restore the strength of inlay-cemented premolars. Selective enamel acid etching with or without universal adhesive significantly increased the fracture resistance.

Clinical significance: Selective enamel acid etching is recommended for increasing the fracture resistance of the SA cemented composite inlay to the level of intact teeth.

Keywords: Acid-etching, Fracture resistance, Inlay, Self-adhesive cement, Universal adhesive.

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Introduction

Adhesive restorations are known to strengthen the weakened tooth structure following cavity preparation with the removal of marginal ridges.[1] This reinforcement associated with well-adapted and sealed margins could guarantee long-lasting restorations in damaged teeth.[2] An indirect approach is preferred to direct composite resin, especially in wide cavities in terms of marginal sealing due to minimized impact of polymerization shrinkage, improved physical/mechanical properties, and also simply producing correct proximal contacts and contours.[2–4] The advantages of composite resins compared to ceramics have resulted in their widespread use as intracoronal restorations. Composite resins exhibit less abrasive effects on the opposing tooth and greater fatigue/fracture resistance, especially during try-in.[4,5] The lower the elastic modulus, the better the stress distribution and the better the bonding between composite resin and tooth structure and luting resin cement could contribute to a higher fracture resistance of the restored teeth, creating a monoblock restoration and reinforcing the restoration.[3,5,6] Despite involvement of the two adhesive interfaces, the weakest one determines the final bond strength.[2] Different treatments providing sufficient surface activation/roughness result in chemical bond and mechanical interlocking at the cement–tooth structure interface.[2] Adhesive systems in two types, E and R and self-etch (SE), are recommended to increase bonding of the resin cement to cavity walls and reinforce the restored teeth.[7] However, SA resin cements are applied without the adhesive system. This is associated with lower technique sensitivity, simplified application and short cementation time; hence, they are attractive in clinical practice.[8] However, the efficacy of adhesive bonding compared to those of E and R or SE cements has been reported with conflicting results.[7,9–11] Despite initial acidity due to acidic monomers, the high viscosity of SA cement and low etching ability along with buffering effect of enamel/dentin minerals and lack of smear layer removal contribute to superficial interaction with dental structure, resulting in low bond strength.[8,12,13] This low bonding capacity could compromise the strength of SA-cemented restored teeth.[14] A number of surface treatments have been evaluated to improve the bonding ability of SA cements.[7,11] Some of them were reported to be successful, with others being unsuccessful, depending on the brand of SA cements.[5,16–20] However, no study has examined the effect of these treatments on the strength of restored teeth. Consequently, this study was designed to test the null hypothesis stating that different treatments in a cavity prepared for inlay have no impact on FR of premolars with SA-cemented inlay.

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Materials and Methods

Following the approval of the research protocol by the Shiraz University of Medical Sciences Ethics Committee, 120 maxillary single-rooted premolars, extracted for orthodontic reasons, were selected. The teeth were intact with no defect and fracture or crack lines as verified under ×20 magnification. The samples were cleaned and disinfected in 0.5% chloramine solution and then stored in distilled water at 4°C. The buccopalatal and mesiodistal dimensions of the teeth, measured with a digital caliper (Mitutoyo Digimatic; Mitutoyo, Kawasaki, Japan), were 9 and 7 mm, respectively, with a variation of 0.5 mm for each dimension. Prior to embedding the teeth in a cylinder of self-curing acrylic resin up to 1 mm below the cementoenamel junction (CEJ), their roots were covered with a 0.2–0.3 mm layer of melted wax. This layer was replaced with a polyether impression material to mimic the periodontal ligament.[21] The long axis of the tooth was perpendicular to the base of the cylinder. The teeth were randomly separated in 10 groups ($n = 12$). Group I (intact): the intact teeth served as a negative control. The other teeth were subjected to inlay preparation.

MOD Inlay Preparation

Standardized MOD cavities were prepared with conical round-ended diamond burs (#7875, Teeskavan, Iran) in a high-speed handpiece under water and air cooling. The preparations had round internal angles, 6° divergent walls, and an occlusal box with a width of two-thirds of the intercuspal distance and a buccopalatal dimension of 3.5 ± 0.2 mm. The cervical wall was placed 1 mm above the CEJ in enamel, with a depth of 4 ± 0.2 mm at the isthmus. The preparations had only buccal and palatal walls, with no axial walls. The diamond bur was replaced after every five preparations.

Group II (prep): The prepared teeth were not restored and served as a positive control.

Inlay Restorative Procedures

Following the isolation of the cavity surfaces with a medium of water-soluble gel (Johnson and Johnson, New Brunswick, NJ, USA), the composite inlays were fabricated with Z250 (3M ESPE, St. Paul, MN, USA) using the oblique incremental technique. Light curing was carried out with a halogen light unit (Coltolux, Coltene Whaledent, Attstatten, Switzerland) at a light intensity of 500 mW/cm^2. The light intensity output was checked every five restorations with a radiometer from the same manufacturer. The composite inlays were then removed from the cavity and further polymerized in an oven at 100°C for 10 minutes. After air-particle abrasion of the internal surfaces of inlays with 50-μm alumina particles (Microetcher, Dento-Prep, Ronving, Denmark), washing and air drying, a silane agent (VOCO, Cuxhaven, Germany) and then a layer of Solobond M (VOCO) were applied and light cured for 20 seconds.

The inlays were cemented in groups III–X. In group III (E and R/Con), the cavity surfaces were etched with 35% phosphoric acid for 15 seconds. After rinsing for 15 seconds and gentle air drying, Solobond M (VOCO) was applied and light cured for 20 seconds. Two pastes (base and catalyst) of the conventional resin cement (Bifix QM, VOCO) were mixed through self-mixing tip and inserted on the surfaces of the cavity and inlay. The inlay was cemented under 1 kg seating load for 5 minutes; after removing the excess cement with a microbrush, light curing was performed for 40 seconds from each side of the tooth.

In group IV (SA), the mixed SA cement, Bifix SE (VOCO), was applied to the cavity and inlay surfaces by means of a self-mixing tip and the inlay was cemented similar to that in group III.

In groups V–X, the inlay was cemented with Bifix SE as described in group IV, following different cavity pretreatments as follows:

In group V (EDPA/SA), the enamel and dentin surfaces were etched with 35% phosphoric acid for 15 seconds. After rinsing and gentle air-drying, SA cementation was performed.

In group VI (EPA/SA), only the enamel surfaces were acid etched for 15 seconds.

In group VII (SEUA/SA), after acid etching only the enamel surface, a universal adhesive, Futurabond U (VOCO), was applied on the enamel and dentin surfaces for 20 seconds, followed by gentle air drying and light curing for 20 seconds. This served as a selective enamel-etching approach.

In group VIII (ERUA/SA), after acid etching the enamel and dentin surfaces, Futurabond U was applied similar to that in group VIII, as an etch-and-rise approach.

In group IX (EDTA/SA), the enamel and dentin surfaces were conditioned with 17% EDTA (Master-dent, Dentonics, Inc. USA) for 60 seconds and rinsed for 30 seconds and gently air dried.

In group X (polyacrylic acid (PAA)/SA), the enamel and dentin surfaces were conditioned with 20% polyacrylic acid (Cavity Conditioner, GC, Tokyo, Japan) for 10 seconds, rinsed for 20 seconds, and gently air dried.

The cemented inlays were finished, polished, and stored in distilled water at 37°C for 1 week. A single operator (N/H) performed all the inlay preparations, fabrication, and cementation. Types, specifications, and manufacturers of the utilized materials are listed in Table 1.

Table 1: Materials' characteristics used in this study

Material/manufacture/ lot no.	*Type*	*Composition*
Bifix SE/VOCO, Cuxhaven, Germany/1714134	SA resin cement	Bis-glycidyl methacrylate (Bis-GMA), aliphatic, aromatic and acid methacrylate, benzoyl peroxide, amines, butylated hydroxytoluene (BHT)
Bifix QM/VOCO, Cuxhaven, Germany/001217	Conventional resin cement	Bis-GMA, benzoyl peroxide, amines, barium–aluminum boro-silicate glass
Futurabond U/VOCO, Cuxhaven, Germany/1550316	Dual-cure universal adhesive	Liquid 1: acidic adhesive monomer, hydroxyethyl-methacrylate (HEMA) Bis-GMA, HEDMA, urethane dimethacrylate (UDMA) catalyst Liquid 2: ethanol Initiator, catalyst
Solobond M/VOCO, Cuxhaven, Germany/1339627	E and R adhesive	Methacrylates, acetone, oromatic and acid derivatives, an organic fluoride component
EDTA/Master-dent, Dentonics, Inc, USA/9515	Conditioning agent	0.5 M EDTA in water
Cavity conditioner/GC, Tokyo, Japan/1402261	Conditioning agent	20% polyacrylic acid, 3% aluminum chloride hexahydrate

Aging Procedures and Fracture Resistance Test

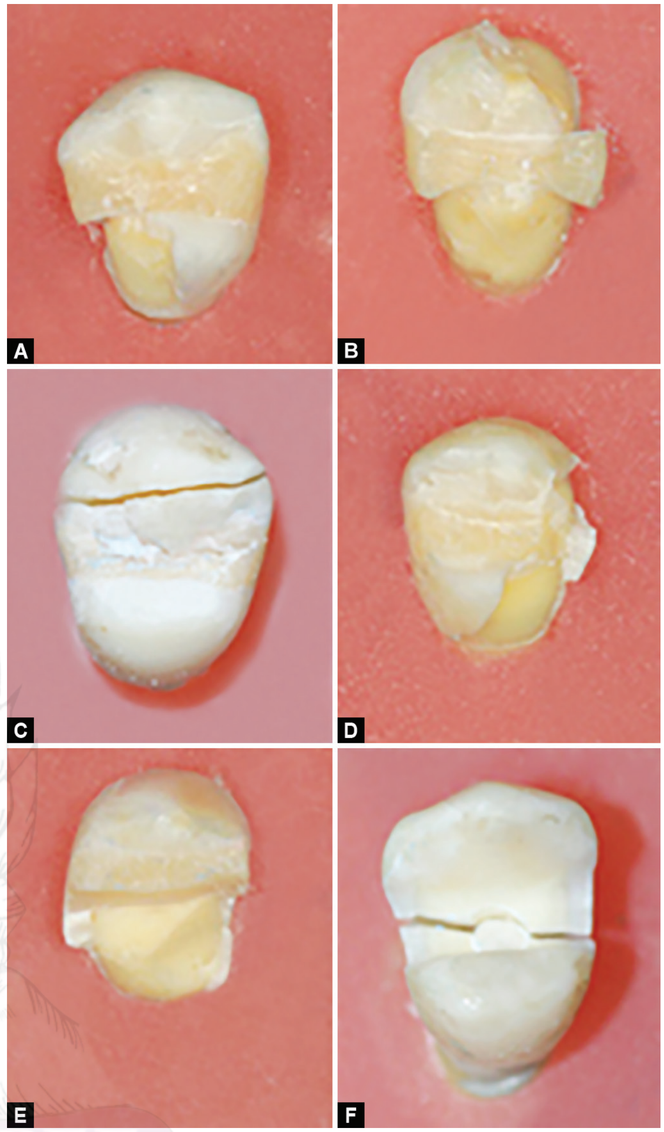
All the specimens were subjected to 100,000 cycles of application of 50 N loading forces at a frequency of 0.5 Hz in a mastication simulation machine (Chewing Stimulator CS4; SD Mechatronic, Feldkirchen, Westerham, Germany).[22] The mechanical load was applied to the center of the occlusal surface in contact with both cusp ridges using a stainless steel antagonist with a rounded end that was 6 mm in diameter in a water environment. After a 6 month water storage period and thermal cycling (Vafaie Inc, Tehran, Iran) for 1000 cycles at 5°C/55°C (dwell time: 15 seconds), the specimens were subjected to a compressive load at a crosshead speed of 1 mm/min in a universal testing machine (Zwick Roell, Ulm, Germany). The compressive load was applied parallel to the long axis of the tooth with a 6 mm diameter stainless steel antagonist placed in the center of the tooth with contacts only on the buccal and palatal cuspal inclines. The peak force required for fracture was recorded in Newton as the fracture strength (FR) value.

Data were analyzed with the normality test (Kolmogorov-Smirnov test), verifying lack of normal distribution. Therefore, data were analyzed with one-way ANOVA and Dunn tests ($\alpha = 0.05$).

Fracture Mode Evaluation

After FR testing, the specimens were assessed to classify the fracture modes as follows:

- Mode I: Cusp fracture extending to CEJ
- Mode II: Cusp fracture extending below the CEJ or fracture at the cusp–inlay interface
- Mode III: Partial restoration fracture along with cusp fracture at the CEJ
- Mode IV: Partial restoration fracture along with cusp fracture extending below the CEJ
- Mode V: Longitudinal fracture dividing the tooth along the axis (Fig. 1)



Figs 1A to F: Different types of fracture: (A) Mode I, cusp fracture extending to CEJ; (B) Mode II, cusp fracture extending below the CEJ; (C) Mode II, cusp fracture at the cusp–inlay interface; (D) Mode III, partial restoration fracture along with cusp fracture at the CEJ; (E) Mode IV, partial restoration fracture along with cusp fracture extending below the CEJ; (F) Mode V, longitudinal fracture dividing the tooth along the axis

Results

Fracture resistance values in Newton (median, mean ± SD) for the 10 groups are presented in Table 2.

A statistical comparison of FR data of the study groups revealed significant differences between them ($p < 0.001$). Among the experimental groups, group VII (805 N), group III (785 N), and then group VI (775 N) revealed the highest and comparable FR, with no significant difference from group I (1025.5 N) ($p > 0.05$) but with significant differences from the other groups ($p < 0.05$). In group IV (500 N) and group V (435 N), the second highest and comparable FRs were obtained, which were not significantly different from group VIII (411 N), group XI (397 N), and group II (311 N), but significantly higher than that in group X (312) ($p = 0.02$). The latter group had the lowest FR with a significant difference from other groups ($p < 0.02$), except for groups VIII, IX, and II.

In most of the groups, mode I and mode II fracture patterns were the predominant modes, except for the intact group in which all the fracture patterns consisted of mode I.

Discussion

This study evaluated the effect of cavity pretreatments on FR of premolars with composite inlay cemented using the SA cement. A number of studies on the effects of pretreatments on the efficacy of SA cements were all dentin/enamel bond strength assessment;[7,9–11,15–20] they have some deficiencies in relation to clinical situation. These tests were performed on flat small surface areas of tooth structure; therefore, the effects of more complex inlay cavity, the relevant C-factor, and compliance of cavity design were not involved.[7] Moreover, seating force during cementation process that might overcome the high viscosity/low penetration of the SA cement was not applied. The flat bonding surface is abraded using 600-grit silicon carbide to standardize the smear layer. However, this procedure cannot mimic the clinical situation since bur-prepared dentin surface of inlay cavity is composed of thicker and more compact smear layer.[23,24] This might impede bonding interaction of SA and mild SE cements.[24] Therefore, the FR test is thought to favorably provide the possibility of simulating clinical conditions and chewing cycles on restored teeth.

Table 2: Fracture resistance in Newton (median and mean ± SD) and fracture mode in the 10 study groups ($n = 12$)

Groups	*Median**	*Mean ± SD*	*Fracture mode•*
Group I	1025.5^A	1046.6 ± 138	12/0/0/0/0
Group II	311.0^BC	370.3 ± 132	10/2/0/0/0
Group III	785.0^A	745.1 ± 203	5/5/1/1/0
Group IV	500.0^B	520.7 ± 154	5/6/1/0/0
Group V	435.0^B	505.0 ± 134	4/7/0/1/0
Group VI	775.0^A	748.0 ± 122	5/6/1/0/0
Group VII	805.0^A	792.9 ± 181	4/5/3/0/0
Group VIII	411.0^BC	466.1 ± 136	4/5/1/2/0
Group IX	397.0^BC	404.0 ± 104	3/5/1/2/1
Group X	312.5^C	315.0 ± 90	2/5/1/3/1

*Medians followed by the same superscript letter did not differ statistically significantly according to the Dunn test at a significance level of 5%
•Mode I, cusp fracture extending to the CEJ; mode II, cusp fracture extending below the CEJ or at the cusp-inlay interface; mode III, partial restoration fracture along with cusp fracture at the CEJ; mode IV, partial restoration fracture along with cusp fracture extending below the CEJ; mode V, longitudinal fracture dividing the tooth along the axis

Adhesive cementation could increase the strength of premolars with MOD cavities.[1,14] However, SA cement used in this study did not verify this beneficial effect. Also, various treatments prior to the SA cementation affected the FR of the inlay-restored premolars differently, rejecting the tested null hypothesis.

In the current study, FR was tested after chewing simulation, thermal cycling, and water storage. Using variable numbers of repeated subcritical load in the range of normal biting/chewing force prior to ramped loading to fracture could not induce acute failure, but it had a negative effect on FR through crack initiation/propagation within the restored teeth.[25] The thermal and water aging might have weakened the adhesive bonding.[26] These aging processes could reduce reinforcing capacity of adhesive-bonded inlay. This bonding reduction might be different among various adhesive approaches. In light of our results, E and R adhesive along with conventional resin cement and selective enamel etching with or without universal adhesive, among different treatments prior to SA cement, were able to somewhat restore the strength of the inlay-restored teeth to the level of intact teeth, whereas SA cement alone could not reach it. This is in agreement with the results of a study by Sallaverry et al.[14] However, enamel and dentin acid etching with or without the use of Futurabond U did not exhibit this ability. This finding could support the idea that in the case of SA cementing, enamel acid etching only was capable of restoring the strength of the teeth, while acid-etching dentin with or without subsequent application of Futurabond U resulted in no beneficial effect on the FR.

Despite consistent promising results of bond strength studies of SA cements on acid-etched enamel, convergent results on the effect of dentin acid etching on bond strength, from beneficial effect to adverse and no effect, have been reported.[7,8,11,12,16,27–30] It appears that this effect has been product specific. They differ substantially in various properties, including chemical composition, physical properties, pH, setting reaction, and viscosity. Hence, they are not considered as a unity.[9,19] Although smear layer removal and dentin demineralization facilitated penetration of acidic monomers of SA cement, especially under seating pressure, lack of minerals excluded the chemical interaction of the acidic monomers.[16] On the contrary, the high viscosity of the cement might impede its infiltration into a thick and compact exposed collagen network of acid-etched dentin, leaving the nonresin-impregnated layer vulnerable to degradation process.[7,12] Moreover, the pressure during seating of inlay might lead to collagen matrix collapse.[8] The use of a low-viscosity adhesive could wet/infiltrate better than that performed by SA cement on etched dentin. The association of E and R adhesive with SA cement, especially for the Bifix SE with Solobond M, was reported to increase the short-term dentin bond strength.[15] However, overall, no positive effect of dentin etching with or without adhesive on FR was recorded after aging. During aging, cyclic loading induced degradation of the exposed collagen with no resin impregnation by endogenous proteases in the two dentin-etched groups, especially with no adhesive application.[31] Contrary to E and R mode, SE mode of Futurabond U significantly increased FR of SA-cemented premolars in this study. Adequate bonding durability of the SE mode of universal adhesives has recently been reported.[32] The higher bonding stability of their SE mode was demonstrated compared to E and R mode over time.[33,34] This SE approach was performed along with selective enamel acid etching. This group yielded an FR comparable to that of the EPA/SA group in which only enamel was acid etched and no adhesive was subsequently applied. This finding confirmed the important role of acid-etched enamel bonding in restoring the strength, while for the dentin, SE or SA approach was preferred. Therefore, the lower strengthening effect of SA cementation was related to lower bonding ability to the enamel not to the dentin. Although adequately stable enamel bond in all the margins of the inlay cavity prepared in this study might have limited degradation of dentin-adhesive interface, fatigue loading could have negatively influenced this interface. The similar bonding efficacy of SA and conventional E and R cements to dentin has been reported for some SA cements, not for all of them.[10,35]

In the case of EDTA and PAA pretreatments used in this study, the results were not promising, even for PAA; the performance of SA cementation was considerably lower. In this line, an adverse effect of PAA application on bond strength of the SA resin cement (RelyX U200) to enamel and dentin was reported in a recent study, with the same adverse effect on dentin bonding of another SA cement.[19] However, there are reports of no effect or positive effect on dentin bonding ability of some SA cements by different concentrations of PAA (10–40%).[17,18,36,37] These divergent results depend on different brands of the cement used and their compositions. Although milder etching capacity of PAA and EDTA, compared to acid etching, might be beneficial in terms of dentin bonding, it could not establish durable and strong enamel bonding.[12,18] Phosphoric acid etching of enamel that is a highly mineralized structure compared to that of dentin removes the smear layer and partially demineralizes it. The subsequent surface with high surface energy is more receptive for bonding.[28]

All the products (SA cement, Bifix SE and universal adhesive, Futurabond U) used in the current study were from the same manufacturer. Although the pH of Futurabond U is 2.3, a dual-cured activator containing this two-component adhesive could prevent incompatibility between the cement and acidic adhesive in deep parts of the cavity in which the cement would cure through self-curing reaction.[38,39]

SA cement used with selective enamel-etching with or without Futurabond U exhibited FR in the level of E and R/Con cement. The use of SA or SE approach in deep dentin of the inlay cavity is thought to provide more suitable and effective bond compared to the E and R approach because of not complete removal of the smear layer. In addition, this cementation approach could help reduce post-cementation sensitivity that is often observed with E and R cementation. SA cement/selective enamel etching could be also considered a simplified, time-saving procedure. Although static loading used in FR test might not have clinical relevance, it was demonstrated to be a valid method to compare adhesive restorative materials. Fatigue loading might produce better normal intraoral function. However, the linear relationship between fatigue and static loading was demonstrated.[40,41]

This study was conducted on one product of SA cements. With respect to their various compositions, further studies are required to reach to a final conclusion to answer the question whether an additional surface treatment could be suggested to enhance the SA cementation, while it negate simplified application of SA cements.

The present study had some limitations. All variables of intraoral situations were not included. The pulpal pressure was not simulated and the cemented teeth were not subjected to pH changes and enzymatic challenges that could interfere with the cement–tooth interface.[42,43]

Conclusion

Considering the limitations of this study, it can be stated that

- Contrary to E and R adhesive/conventional resin cementation, SA-cemented inlay was not capable of restoring the strength of MOD-prepared premolars.
- Among different surface pretreatments, only enamel acid etching with or without universal adhesive in the SE mode for dentin surface could provide FR to the level of intact teeth.
- Polyacrylic acid adversely affected the strengthening property of SA cement.

Clinical Significance

Selective enamel acid etching is recommended for increasing the fracture resistance of SA-cemented composite inlay to the level of intact teeth.

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