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Influence of enamel acid-etching on mechanical properties and nanoleakage of resin composite after aging

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Aim: The aim of this study was to evaluate how acid-etching of the cavosurface enamel in Class I resin composite restorations influences the bond strength to the pulpal wall and the restoration, Knoop microhardness and nanoleakage after thermomechanical aging. For this research 76 fresh human molars were selected and restored with Silorane or Clearfil SEBond/Z350XT composite divided in 4 groups (Silorane system restored with or without enamel cavosurface acid-etching and Clearfil SEBond/Z350XT with or without enamel cavosurface acid-etching). To induce artificial aging, samples were subjected to thermomechanical cycling through 200,000 and thermal cycling between 5 and 55 °C with 30 second filling and 15-second drainage steps. Microhardness and microtensile bond strength were evaluated in 32 teeth (n=8) each and nanoleakage evaluation was performed in 12 teeth (n=3). Samples restored by Clearfil SEBond/Z350 XT without cavosurface acid-etching showed significantly lower microtensile bond strength results. The resin composite Z350XT presented higher values of Knoop microhardness. It was observed little or no infiltration for Silorane groups and moderate infiltration for Clearfil SE Bond groups. Acid-etching of the cavosurface enamel during restoration procedure with Clearfil Se Bond resulted in a stronger bond after thermomechanical cycling. Silorane groups showed less infiltration than Clearfil SE Bond groups.

Keywords: Dental cavity preparation; Tensile strength; Silorane Resins; Adhesives.

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Introduction

Shrinkage of dental composites is an ongoing challenge in dentistry. Composite shrinkage can cause adhesive failure and loss of marginal sealing[1], resulting in post operative sensitivity, marginal staining, and secondary caries[2].

Silorane is a low-shrinkage, low-interfacial stress resin composite composed of a matrix of siloxane and oxirane. It is polymerized through a cationic reaction involving the cycloalyfatic oxirane ring opening. The resulting composite exhibits less than 1% shrinkage[3], which leads to better marginal integrity[4] and less microleakage compared to methacrylate composites[5]. In addition to the monomer composition, factors that influence the stresses created by polymerization include the photo initiator, number and type of filler, c-factor, restorative technique, and light source[1,6].

The composites are used with adhesive systems. Compared to total-etch adhesives, self-etch adhesive systems are easier to manipulate, can be applied more rapidly[6], and are less technique-sensitive, leading to fewer errors during application[7]. However, self-etch adhesives can present deficient penetration of the enamel[8], leading to faster degradation of the interface[9-11] and subsequent infiltration of the hybrid layer. Acid-etching of the cavosurface enamel has been suggested as a means to improve the bond strength of the enamel, as well as the longevity and quality of the interface[12,13]. However, the vector of polymerization shrinkage is directed towards the walls with the highest bond strength. An overly large increase of the enamel bond strength could cause a decrease of the dentin bond strength, even when a layering technique is used, because the stress of polymerization shrinkage would be concentrated on this area[14].

In clinical evaluation, the microhardness of a composite material is an important indirect means for estimating the degree of conversion (DC) of the polymer[15]. A polymer that presents a relatively low DC will exert less stress after polymerization shrinkage. Although this condition leads to better marginal stability, it could also masks the bond strength results.

In this study, the Knoop microhardness was tested to evaluate the DC and to ascertain (indirectly) the stress at the restoration interface. The aim was to evaluate how acid-etching of the cavosurface enamel during a Class I restoration influences the Knoop microhardness of the composite, the microtensile bond strength between the pulpal wall and the restoration composite, and the nanoleakage after thermomechanical aging. The hypotheses of this study were as follows: (1) acid-etching would influence the quality of the bond strength on the pulpal wall/restoration after aging, (2) Silorane would present superior mechanical properties, and (3) Nanoleakage would not differ between the two tested restorative systems.

Material and Methods

This research was approved by the Ethics Research Committee of Piracicaba Dental School – University of Campinas (number N-089/2012). The study consisted of three tests, measuring the microtensile bond strength, the microhardness, and the nanoleakage. The materials used are described in Table 1.

Table 1. Composition and manufacturers of materials used on this study.

Material	Composition	Manufacturer
Scotchbond acid	• phosforic acid; -water; • poli(éter vinil).	3M ESPE, St. Paul, Minnesota, USA
Silorane Primer	• 2-hidroxiethil metacrilate (HEMA); • Bisphenol-a-diglicidil éter dimetacrilate (BIS-GMA); • water; -etanol; -silic treated with silane; • phosforic acid-metaciloxic-hexilester; • 1.6 hexanodiol dimetacrilate; • copolimer acrilic and itaconic acid; (dimetalimine) etil metacrilate; • DL-Canforoquinone; -Phosfine acid.	3M ESPE, St. Paul, Minnesota, USA
Silorane Bond	• Dimetacrilate; -Silic treated with silane; • Trietilene glycol dimetacrilate (TEGDMA); • Phosforic metacriloxi-hexilesters acid; • DL-Canforouinone; • 1.6- hexanodiol dimetacrilate	3M ESPE, St. Paul, Minnesota, USA
Silorane Composite Filtek P90 A3	• 3,4- epoxiciclo hexiletilciclopolimetil siloxane; • bis-3,4 – epóxi ciclohexiletilfenilmetil silane; • Silanizaded quartz; -Itriumfluoride; • Canforoquinone	(3M ESPE, St. Paul, Minnesota, USA
Clearfil SE Bond Primer	• 10-Metacriloiloxi -decil dihidrogenade fosfatase (MDP); • HEMA; -Dimetacrilate hidrofílic; • Canforoquinone; -Terciary amine; -Water	Kuraray Medical Inc. Okayama, Japan
Clearfil SE Bond Bond	• HEMA; -10-Metacriloiloxi -decil dihidrogenado fosfatase (MDP); • Bis-GMA; -Dimetacrilate Hidrofílic; • Terciary amine; • Sílic Coloidal silanizaded; -Canforoquinone.	Kuraray Medical Inc. Okayama, Japan
Composite Filtek Z350 XT A3	• Bis-GMA; -Bis-EMA6; • UDMA; -TEGDMA; -Silic; -Canforoquinone	(3M ESPE, St. Paul, Minnesota, USA

Briefly, 76 freshly extracted human third molars were stored for 24 hours in a buffered 0.1% thymol solution at 37 °C. After cleaning, the teeth were stored in distilled water until cavity preparation. The teeth were embedded in polystyrene resin. Their occlusal surfaces were planed in a polishing machine (Arotec Ind., São Paulo, Brazil) with 400-grit sandpaper (3M 411Q, Sumaré Brazil, SP, Brazil). A standard cavity preparation machine was used to create a Class I cavity in each polished tooth. Each cavity had the following dimensions: 5 mm in the mesiodistal direction, 4 mm in the buccolingual direction, and 3 mm in depth. Cavities were made with a #56 carbide bur (KG Sorensen Ind. E Com Ltda., Barueri, SP, Brazil), which was replaced after every 5 cavities.

For the restorative procedure, half of the samples (38 teeth) were randomly selected and subjected to acid-etching of cavosurface enamel with 35% phosphoric acid for 30 seconds. The surface was washed thoroughly with water for 30 seconds and dried with air jets. Half of these acid-etched teeth (19 teeth) were restored with Silorane Systems (3M ESPE, St. Paul, Minnesota, USA) (CAPN) and the other half (19 teeth) with Clearfil SE Bond (Kuraray Medical Inc. Okayama, Japan)/Z350 XT (3M ESPE, St. Paul, Minnesota, USA) (CACZ). Similarly, for the samples that had not been acid-etched (38 teeth), 19 teeth were restored with Silorane Systems (SAPN) and 19 teeth with Clearfil SE Bond/Z350 XT (SACZ). The restorative procedure is described in Table 2.

After being kept in an environment at 37 °C with relative humidity for 24 hours, samples were placed in a thermomechanical cycling machine (MSFT, Elquip, São

Table 2. List of the groups of this study where CA = presence of acid etching and SA = absence of acid etching. PN = Silorane and CZ = Clearfil SE Bond/Z350

Group	Enamel Acid etching	Restorative Systems	Restorative Procedures
CAPN	PRESENT	Silorane Systems	After drying with air jets, actively primer was applied to enamel and dentin for 15 seconds with a microbrush, gentle air for 10 seconds of 10 centimeters apart and curing for 10 seconds. Then the adhesive was applied with a microbrush across the cavity and gentle air jet for 10 seconds to 10 centimeters apart and curing for 10 seconds with halogen light unit QTH Lamp (Bisco, Schaumburg, Illinois, USA). The teeth were restored with Silorane composite resin in six increments cured for 40 seconds each with the same unit.
SAPN	ABSENCE	Silorane Systems	
CACZ	PRESENT	Clearfil SE Bond/Z350XT	It was actively applied the primer with a microbrush on enamel and dentin for 20 seconds and dried with gentle air for 10 seconds to 10 centimeters distance. Then applied an even layer of adhesive to enamel and dentin with a microbrush for 20 seconds and cured for 10 seconds. The teeth were restored with composite resin Z350XT in six increments and light cured for 40 seconds each with the same halogen light system used on Silorane composite groups.
SACZ	ABSENCE	Clearfil SE Bond/Z350XT	

Carlos, SP, Brazil). Each sample underwent 200,000 cycles of loading at 80N and 2 cycles/second. Loading was applied perpendicular to and in the centre of the restoration. Thermal cycling was performed by 30-second water filling and 15-second drainage steps, with temperatures ranging between 5 and 55 °C.

Microhardness and microtensile bond strength were evaluated in 32 teeth (n=8) each. Nanoleakage evaluation was performed in 12 teeth (n=3) (Figure 1). For the microtensile bond strength test, dental crowns were separated from the root portion, perpendicular to the long-axis of the tooth, with a double-sided diamond disc (KG Sorensen). The crowns were set in a metallographic precision cutter (Isomet 1000, Buehler Ltd., Lake Buff, IL, USA). Serial sections perpendicular to the long-axis of the crowns were cut with a high-concentration diamond disc (Extec Corp., Enfield, CT, USA), used at low speed and under constant irrigation. This process resulted in stick-shaped samples (0.9 × 0.9 mm), where each stick included a portion of the bonding interface to the pulpal wall. The sticks were kept in an environment with relative humidity until the microtensile test. The fracture mode of each sample was evaluated on scanning electron microscopy (SEM) and classified as adhesive, mixed, or cohesive in dentin or resin.

For the Knoop microhardness test, the crowns were separated from the root portions of the teeth as described above. The crowns were placed in a metallographic cutter and sliced into two parts through the centre of the restoration, in the mesiodistal direction parallel to the long-axis of the tooth. Both parts of the tooth were embedded in the same cylinder of polystyrene resin, to facilitate microhardness testing. The restoration was finished with silicon carbide sandpaper (600-, 1200-, and 2000-grit) and polished by diamond paste (3-, 1-, and 0.25-mm granulations), applied with felt disks on a polisher underwater cooling. Between each sandpaper treatment, the samples were ultrasonicated in distilled water for 5 minutes. To measure the microhardness, three indentations were made for 20 seconds each under a 25g load (HMV-2000,

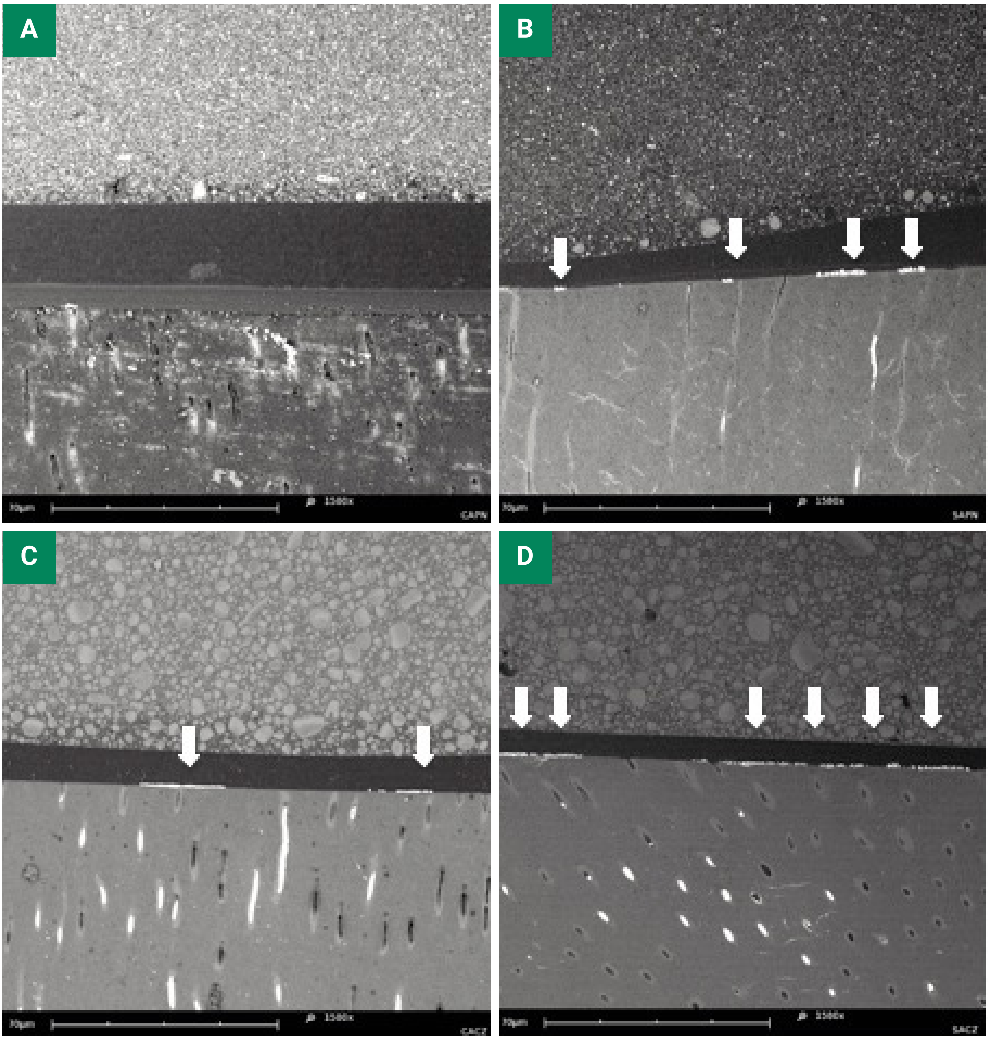

Figure. A- SEM Photomicrography showing no silver deposits for the Silorane systems with previous acid-etching; B- SEM Photomicrography for the Silorane systems without acid etching (arrows show a little infiltration of silver nitrate); C- Nanoleakage for the group Clearfil/Z350 XT with acid etching (arrows showing the silver nitrate moderate infiltration in the hybrid layer); D- Nanoleakage for the group Clearfil/Z350 XT without acid etching (arrows showing the silver nitrate moderate infiltration in the hybrid layer).

Shimadzu, Japan) at three depths measured from the top of the tooth: 100 µm (top), 1500 µm (middle), and 2900 µm (base), at 25 µm from the mesial or distal wall.

Nanoleakage analysis was performed on 12 teeth (n=3). Using a metallographic cutter machine, three mesiodistal cuts were made in each tooth, resulting in two 1-mm-thickslices. Slices were immersed in a solution of ammoniacal silver nitrate for 24 hours, washed in distilled water, immersed in light developer for 8 hours, and then embedded in polystyrene resin[16]. Embedded samples were polished with silicon carbide sandpaper and diamond paste (3-, 1-, and 0.25-µm granulations). Samples were demineralized and deproteinated with 85% phosphoric acid and 2% hypochlorite.

Samples were dehydrated in serial ethanol solutions (25%, 50%, 75%, 90%, and 100%). Dehydration was maintained by silica until the samples were ready to be coated with carbon (Baltec SputterCoater - SCD – 050) for viewing by Phenom Microscope (Dillenburgstraat 9E – Netherland).

Results

Microtensile test results

Etching (p = 0.0015) and the interaction between the restorative system and etching had significant effects according to ANOVA two-way. Therefore, the post-hoc Tukey test was applied for interaction (Table 3), which revealed that samples restored by Clearfil SE Bond/Z350 without cavosurface enamel acid-etching had the lowest microtensile bond strength results among the groups ($p < 0.05$) (Table 3). No other significant differences in microtensile bond strength were observed.

All of the samples exhibited a mixed fracture pattern, with the adhesive showing the largest fracture areas and fractures also observed in the dentin.

Table 3. Results of Tukey test to multiple comparations for microtensile test.

GROUP	MEAN (MPa)/TUKEY
CACZ	25,66 (±5,62)a
SAPN	22,69 (±5,52)a
CAPN	21,71 (±5,78)a
SACZ	12,16 (±4,33)b

Knoop hardness test

For the microhardness test results, ANOVA two-way only revealed statistically significant differences for the composite resin (p = 0.0001) and the interaction between the composite and cavosurface acid-etching (p = 0.0109). Interactions between other factors or interactions had no statistical significance. Therefore, the post-hoc Tukey test was applied to the interaction between the composite and cavosurface acid-etching (Table 4), which showed significant differences in microhardness between the Z350 XT and Silorane composite resins, independent of whether cavosurface acid-etching was performed and the depth at which the microhardness was measured.

Table 4. Results of Knoop hardness test (KHN) for composite x cavosuperficial enamel etching.

GROUP	MEAN (KHN)/TUKEY
SACZ	96,398 (±3,54)a
CACZ	95,152 (±6,76)a
CAPN	66,613 (±6,10)b
SAPN	64,194(±4,63)b

Nanoleakage results

All of the samples were analysed for silver infiltration via Phenom Microscope. Samples restored with Silorane Systems and Clearfil SE Bond/Z350XT with previous acid-etching showed low infiltration (Figure 1a,b e c) of silver nitrate, whereas samples restored with Clearfil SE Bond/Z350XT without previous acid-etching showed moderate infiltration (Figure 1d).

Discussion

The first hypothesis of this study, that acid-etching would influence the quality of the bond strength on the pulpal wall, was found to be partially correct. Statistically lower microtensile bond strength results were observed only for the groups restored with Clearfil SE Bond/Z350 XT without acid etching. The two-step self-etching adhesive system has good clinical performance when the adhesive is bound to dentin, but not to enamel[6,17]. Selective etching has been shown to increase the bond strength of enamel[18], improve the marginal sealing, and reduce the incidence of cracks[19]. Acid-etching of the substrate increases the longevity of the restoration, by reducing degradation of the tooth/restoration interface, compensating for the characteristically poor penetration of the adhesive into the enamel[12,13], and reducing the infiltration into and degradation of the internal walls. These previous findings are consistent with our observation that the bond strength of dentin after artificial aging was reduced in the absence of acid-etching of the cavosurface enamel.

The study of the restoration under simulated aging showed that enamel etching was necessary for optimal use of the Clearfil SE Bond, to prevent the hybrid layer from degrading. Clearfil SE Bond can form a dense polymer network that imposes a certain resistance to water penetration[20]. Compared to other adhesive systems, Clearfil SE Bond reportedly provides satisfactory wettability in smear layer-covered dentin and the lowest contact angle in smear layer-free dentin[21]. Nevertheless, these properties were not sufficient to promote good marginal sealing after artificial aging without acid-etching, probably due to lower interfacial stability caused by infiltration of the hybrid layer.

In contrast, the Silorane adhesive composite showed no difference in behaviour regardless of whether the enamel was conditioned. This two-step bonding system consists of a separately photoactivated primer and adhesive, which form a hybrid layer of 10 to 20 μm[22]. The primer and the bond exhibit conversions of over 90% and nearly 70%, respectively[23]. These high DCs lead to a more robust polymeric structure compared to partially polymerized adhesives, which are more permeable to fluid movement[24]. Although Clearfil SE Bond also has a high DC[23], the highly hydrophobic nature of the Silorane bond likely provides better sealing to dentin[25] and creates an interface that is less prone to weakening[26]. The thick hybrid layer formed by the Silorane adhesive system may act as an elastic buffer[27], which would compensate for the polymerization shrinkage of the composite and the stress generated by artificial aging. Due to its thinner hybrid layer and lower elasticity, Clearfil SE Bond is less able to resist these stresses.

The second hypothesis of this study, that Silorane would present superior mechanical properties, was rejected. The Silorane restorative system showed significantly lower microhardness values than the Clearfil/Z350 XT system, regardless of enamel etching and the depth at which the microhardness was tested. The difference between the composites is mainly due to the type and amount of filler and the composition of the organic matrix[28]. The Silorane composite resin has fewer filler particles[22,28], which are irregular in shape but consistent in size, with a homogeneous distribution[29], which explains the lower values of hardness. In contrast, the Z350 XT composite has higher hardness due to its high content of inorganic nanoparticulate filler[29], which undergoes strong intermolecular interactions within the monomer mixture[30].

Our third hypothesis, that nanoleakage would not differ between the two tested restorative systems, was rejected. These images showed little infiltration of silver nitrate in the Silorane restorative system and Clearfil/Z350 XT group with previous acid-etching, but moderate infiltration in the Clearfil/Z350 XT group without acid-etching. Infiltration of silver ions in the latter group can be explained by the artificial aging, which increased the degradation of the hybrid layer. In contrast, the previous acid-etching for Clearfil/Z350 XT system promote a good marginal sealing, which prevented the infiltration of hybrid layer and increased the interfacial stability. For Silorane system, the elastic buffering effect[27] of the thick hybrid layer may have protected it from artificial aging, lowering the degradation.

Conclusion

Whereas acid-etching of the enamel substrate did not influence the behaviour of the Silorane restorative system, it did increase the microtensile bond strength of the Clearfil SE Plus/Z350 XT restorative system, improving its performance under thermo mechanical aging. The Z350 XT composite resin also had greater hardness than the Silorane composite resin. Silorane restorative system and Clearfil SE Bond/Z350 XT with previous acid-etching showed little infiltration whereas Clearfil SE Bond/Z350 XT without acid-etching showed moderate infiltration within the hybrid layer.

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