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Original Article

In-vitro comparison of the effect of different bonding strategies on the micro-shear bond strength of a silorane-based composite resin to dentin

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ABSTRACT

Background: The current study evaluated the micro-shear bond strengths of a new low-shrinkage composite resin to dentin.

Materials and Methods: In this in-vitro study, 70 extracted premolars were assigned to one of seven groups (n = 10): Group 1: OptiBond Solo Plus (Opt; Kerr); Group 2: SE Bond (SE; Kuraray); Group 3: Silorane System Adhesive (SSA; 3M ESPE); Group 4: OptiBond Solo Plus + LS Bond (Opt LS); Group 5: SE Bond + LS Bond (SE LS); Group 6: OptiBond Solo Plus (Opt Po); and Group 7: SE Bond (SE Po). Occlusal dentin was exposed and restored with Filtek LS (3M ESPE) in groups 1 to 5 and Point 4 (Kerr) in groups 6 and 7. After thermocycling (1000 cycles at 5/55°C), micro-shear bond test was carried out to measure the bond strengths. The results were submitted to analysis of variance and *post hoc* Tukey tests (P < 0.05).

Results: Two-way ANOVA showed no significant differences between the two types of composite resin (P = 0.187), between bonding agents (P = 0.06) and between composite resin and bonding agents (P = 0.894). Because P value of bonding agents was near the significance level, one-way ANOVA was used separately between the two composite groups. This analysis showed significant differences between silorane composite resin groups (P = 0.045) and Tukey test showed a significant difference between Groups 4 and 5 (P = 0.03).

Conclusion: The application of total-etch and self-etch methacrylate-based adhesives with and without use of a hydrophobic resin coating resulted in acceptable bond strengths.

Key Words: Low, contraction, composite resins, bonding agents, micro, shear, bond, strength

INTRODUCTION

Since shrinkage stress has been documented as a causative factor for marginal discrepancies observed in composite resin restorations, considerable efforts have been made to minimize the shrinkage polymerization of composite materials by changing the filler content and shape[1,2] or changing the monomer structure or its chemistry.[3-5]

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Recently, a new category of resin matrix for dental composite resins has been developed based on ring-opening monomers. This composite resin, referred to assilorane, is obtained from the reaction of oxirane and siloxane molecules.[3,4] The advantages of this new restorative material are low shrinkage,[6-9] higher hydrolytic stability[10,11] and mechanical properties, compared to methacrylate-based composite resins (MBC).[3,7,12]

In recent years, 3M ESPE Company has marketed a silorane-based composite resin (Filteksilorane) with its special adhesive (silorane system adhesive). The primer (pH = 2.7) of this adhesive system is hydrophilic because it contains HEMA, which needs to be separately light-cured. Then the hydrophobic viscous resin coating layer (LS Bond) is applied and light-cured.[13]

High concentration of HEMA in the self-etch primer keeps this solution homogeneous and prevents phase-separation effects like those documented for HEMA-poor/free one-step adhesives. Due to the presence of HEMA in hydrophilic primer layer, the resultant bond would be very susceptible to water sorption, leading to a defective polymerization reaction and less desirable curing. Separate application and light-curing of the hydrophobic bond layer is beneficial to bond stability and long-term bond durability. Actually, it seals the dentin surface by blocking osmotic water absorption from dentin.[13,14]

Despite adequate microtensile bond strength, these adhesives show unacceptable nanoleakage, especially between interface of the SSA-primer layer and the SSA-bond layer. In addition, within the SSA-primer itself, some nanoleakage is observed. Both phenomena are related to the hydrophilicity of the SSA-primer that contains HEMA and water which is difficult to remove from the adhesive layer before curing by air-drying. Microtensile tests have shown failure at primer-bonding agent interface instead of the real interface of dentin–adhesive.[13,15] Thus, it can be claimed that the weak site of this adhesive is primer-bonding agent interface.

The manufacturer claims that the choice of one-step self-etch adhesive is based on more popularity of this category of adhesives. However, one-step self-etch adhesives have limitations when they are bonded to dentin,[16] and their long-term bond is still unknown.[17-19] Some questions remain about the bonding abilities of this special adhesive. Although this new composite resin can form a strong bond with identical materials, its capacity to form bonds with dissimilar materials is still under question. If the silorane composite resin (SBC) can adhere to methacrylate-based adhesive, it is proposed as a new subject about how to improve or at least maintain acceptable bond strengths.[14]

Duarte *et al*.[14] demonstrated that the application of a conventional total-etch methacrylate-based adhesive (Single Bond; 3M ESPE), followed by a hydrophobic resin coating (LS Bond), produces acceptable bond strengths of SBC to dentin. In their study, Single Bond was unable to produce sufficient bond strengths with SBC and all the samples were completely debonded. But the application of Single Bond followed by silorane hydrophobic bond resin resulted in adequate bond strength. As a result, a hydrophobic resin coating layer may be placed on top of a methacrylate adhesive to convert silorane into a universal composite resin.[20-22]

In vitro micro-shear bond strength testing is commonly used to quantitatively analyze and rank the performance of adhesive systems on enamel and dentin.[23-25] It has been proved adequate and effective for evaluation and comparison of different adhesive systems and restorative materials.

The aim of this study was to evaluate micro-shear bond strengths in the dentin hybrid layer of low-shrinkage composite resins with different bonding strategies.

MATERIALS AND METHODS

In this invitro study, 70 freshly extracted caries-free human premolars were selected and stored in 0.02% thymol solution for up to one month after extraction. The teeth were scaled, cleaned and stored in distilled water for 24 h. Middle dentin was exposed by sectioning the crowns parallel to the occlusal surface with a low-speed diamond saw (Isomet 1000, Buehler, lake Bluff, IL, USA), after which a standard smear layer was produced by 600-grit abrasive paper. The teeth were randomly assigned to seven experimental groups. The bonded interface was prepared according to the experimental groups [Table 1]. For preparing the samples for the micro-shear bond strength test, we used the methodology developed by McDonough *et al*. (2002)[26] and Shimada *et al*. (2002).[27]

After completing the bonding step, Filteksilorane composite resin material (Lot #N138846, 3M ESPE,

Table 1: Materials used in the study, their compositions and mode of their applications according to the manufactures' instructions

Group	Material	Composition	Manufacturer	Restorative technique
I: OSP	OptiBond Solo Plus	Bis-GMA, hydroxyethyl methacrylate resin, glycerol dimethacrylate, glycerol phosphate dimethacrylate 2-(ethylhexyl)-4-(dimethylamino) benzoate, butylhydroxytoluene, filler (fumed SiO_2, barium aluminoborosilicate, Na_2SiF_6)	Kerr, Italy Lot #3193030	Apply phosphoric acid (35%) to dentin. Wait 15 s and rinse for 10 s. Blot excess water using cotton pellet. Immediately after blotting, apply 2-3 consecutive coats of OptiBond Solo Plus for 15 s with gentle agitation using fully saturated applicator. Gently air thin for 5 s to evaporate the solvent. Light-polymerize for 10 s
II: CSEb	Clearfil SE-Bond	Primer: Water, 10-methacryloxydecyl dihydrogen phosphate, HEMA, camphorquinone, N, N-diethanol-p-to-luidine, dimethacrylate hydrophobic Bond: MDP, bis-GMA, dimethacrylate hydrophobic, CQ, DET, silanated colloidal silica	Kuraray Osaka Japan Lot #00984A	Apply 1 coat of the self-etch primer of SE Bond for 15 s with gentle agitation using fully saturated applicator. Gently air thin to evaporate the solvent. Then apply the bond to the entire preparation using fully saturated applicator. Gently air thin and Light-polymerized for 10 s
III: SSA	Filtek LS System Adhesive	Primer: Phosphorylated methacrylates, vitrebond copolymer, Bis-GMA, HEMA-water, ethanol, silane-treated silica filler, initiators, and stabilizers Bond: Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, and stabilizers	3MESPE, Seefeld, Germany Lot #N170416 3MESPE, Seefeld, Germany Lot #N170417	Apply 1 coat of the self-etch primer of SSA for 15 s with gentle agitation using fully saturated applicator. Gently air thin to evaporate the solvent and obtain an even film. Light-polymerize for 10 s. Then, apply the bond to the entire preparation using fully saturated applicator. Gently air thin until the Bond is spread to an even film and does not move any longer. Light-polymerized for 10 s
IV: OSP LS	OptiBond Solo Plus + LS Bond			Application of OptiBond Solo Plus such as Group 4. Then, bond layer of SSA (LS Bond) applied and light-polymerized for 10 s
V: CSEb LS	SE Bond+LS Bond			Application of SE Bond such as Group 2. Then, bond layer of SSA (LS bond) applied and light-polymerized for 10 s
VI: OSP Po	OptiBond Solo Plus			Application of OptiBond Solo Plus, such as Group 4
VII: CSEb Po	Clearfil SE Bond			Application of SE Bond, such as Group 2

SSA: Silorane System Adhesive

St Paul, USA) was used in groups 1 to 5 and Point 4 composite resin material (Lot #3447643, Kerr, Italy) was used ingroups 6 and 7 directly to fill Tygon tubes (TygonTubing, Saint-Gobain Performance Plastic, Maime Lakes, FL, USA) with an internal diameter and height of approximately 0.7 mm and 1 mm, respectively. The Tygon tubes containing the composite resin were placed on the dentin and light-cured using an LED (Blue Phase, IvoclarVivadent AGFL – 94941: Lichtenstein) device with a light output not less than 600mW/cm². The groups consisted of the following:

Group 1: OptiBond Solo Plus + Silorane composite resin (Opt)

Group 2: SE Bond + Silorane composite resin (SE)

Group 3: Silorane System Adhesive + Silorane composite resin (SSA)

Group 4: (OptiBond Solo Plus + LS bond) + Silorane composite resin (Opt LS)

Group 5: (SE Bond + LS bond) + Silorane composite resin (SE LS)

Group 6: OptiBond Solo Plus + Point 4 composite resin (Opt Po)

Group 7: SE Bond + Point 4 composite resin (SE Po).

The specimens were stored at room temperature (23 ± 2°C) for 1 hour prior to the removal of the tubes by a sharp scalpel blade and then were tested after 1000 cycles at 5/55°C, with a dwell time of 30 sec. The assembly of the composite resin and dentin was attached to the testing device in a universal testing machine (Dartec, Series TLCLO, England) for micro-shear bond testing. A stainless steel blade, 0.4 mm in thickness, was fixed on the superior part of the universal testing machine and gently adapted at the dentin–composite resin interface. A shear force was applied to each specimen at a crosshead speed of 0.5 mm/min until failure occurred. The micro-shear bond strength data were calculated and expressed in MPa.

The results were statistically analyzed ANOVA and post hoc Tukey tests at $P < 0.05$ using SPSS

11.5statistical software (SPSS Corp, Chicago IL, USA).

Fractured surfaces of the specimens were examined under a stereomicroscope (MBC-10, SF-100b, Lomo, Russia) at ×35 magnification, and the fracture modes were determined.

One specimen from each group was gold-coated with a sputter coater (BAL-TEC SCD-500-Sputter coater, Georgia, USA) and examined under ascanning electron microscope (Philips XLC, Philips, Netherland).

RESULTS

Figure 1 shows the means and standard deviations of micro-shear bond strengthsin different groups. In silorane-based composite resin groups the highest mean was found in group 4 (22.39MPa). The lowest mean was found in group 5 (14.57MPa). With Point 4 composite resin themeans of micro-shear bond strengths of OptiBond Solo Plusand SE Bond were 20.82 and 19.00MPa, respectively.

Two-way ANOVA showed no significant differences between the two types of composite resins (P = 0.187). No significant differences were observed between the bonding groups (P = 0.06). The interaction between the composite resin and bonding agentwas not significant (P = 0.894). Since two-way ANOVA showed a P value of 0.06 in the bonding group and the interaction was not significant, one-way ANOVA was used separately between the two composite groups, which showed a significant difference between silorane-based composite resin groups (P = 0.045); *post hoc* Tukey tests showed this significant difference was between Groups 4 and 5 (P = 0.03). The failure mode frequencies of the debonded specimens are shown in Figure 2. The results of failure mode analysis revealed that adhesive fractures were the most common fracture modes and cohesive fractures in compositeresins were the least commonfailure modes, but differences were not significant.

DISCUSSION

This study showed that total-etch methacrylate-based adhesive (OptiBond Solo Plus) used with SBC provides acceptable micro-shear bond strength. Application of this adhesive with LS Bond increased the bond strength but was not significant. It is interesting to note that the mean micro- shear bond
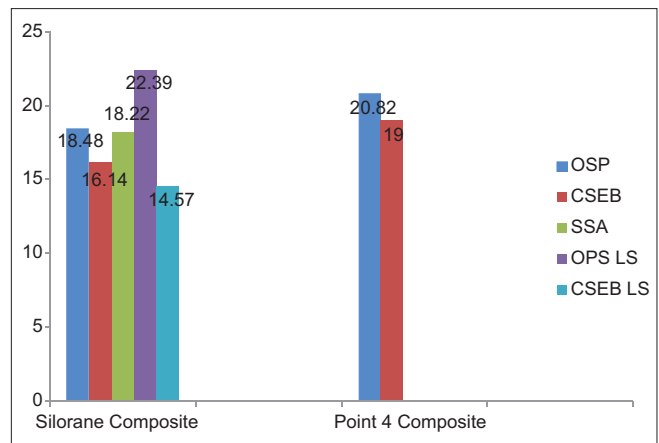
Figure 1: The means and standard deviations of shear bond strengths in different groups. OSP: OptiBond Solo Plus; CSEB: Clearfil SE Bond; SSA: Silorane System Adhesive; LS: LS Bond.
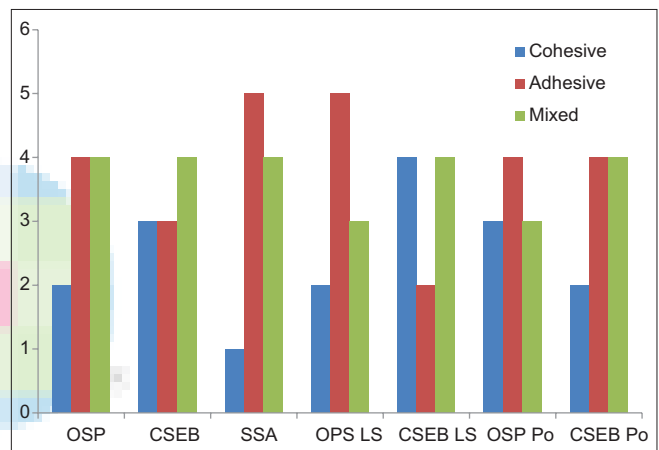
Figure 2: The failure mode frequencies for the micro-shear bond test. OSP: OptiBond Solo Plus; CSEB: Clearfil SE Bond; SSA: Silorane System Adhesive; LS: LS Bond.

strength inGroup 4 (total-etch methacrylate-based adhesive + LS Bond/SBC) was higher than that in Group 3 (special adhesive/SBC).

In the present study, application of self-etch adhesive (SE Bond) with SBC provided adequate micro-shear bond strength, but the use of this adhesive with LS Bond not only increased the bond strength but also decreased it, but this reduction was not significant. No statistically significant difference was observed between bond strength of Opti Bond Solo Plus and SE Bond to Point 4 composite resin and to SBC.

Duarte *et al.*[14] reported that Single Bond was unable to produce sufficient bond strengths with SBC and all the samples were completely debonded. However, the application of Single Bond followed

by LS Bond resulted in adequate bond strength. Nonetheless, in the present research, application of SBC with self-etch and total-etch adhesives resulted in an increase in bond strength. Application of these adhesives followed by LS bond also led to adequate bond strength. One possible explanation for these results could be dissimilarity of conditions in the two studies. In their study, the samples were tested after 24h or after 20,000 thermal cycles and 6 months of aging, whereas in the present study, the samples were tested after 1000 thermal cycles. In addition, the test used in their study (micro-tensile test) was different from that inthe present study (micro-shear test).

SEM evaluations[13,14] have revealed that maximum thickness of the hybrid layer in Silorane System Adhesive is a few hundred nanometers; also SSA has exhibited fewer resin tags. Based on this study, SE Bond had the most frequent resin tags; fewer resin tags were observed for SSA and OptiBondSolo Plus [Figures 3-5] and the resin tags did not influence the bond strengths of one-step self-etch adhesive.[28] However, interlocking of resin monomers with the exposed collagen network of dental structures leads to improvementsin bond strength. Mild and ultra-mild self-etch adhesives hardly demineralize the surface to provide micro-mechanical interlocking,[29,30] but they have the potential for additional primary chemical interaction with hydroxyapatite that remains available on the enamel/dentin surface. This depends largely on the actual functional monomers within the adhesive solution[31] and can explain acceptable bond strength values obtained with these adhesives.

For radical chain polymerizations, oxygen is a powerful inhibitor which retards or even terminates polymerization by reacting with itself or other propagating radicals to form inactive products, resulting in a poorly polymerized resin-rich surface layer.[32] Previous investigations have suggested that the presence of an oxygen-inhibition layer results in no significant increase in incremental bond strength[33,34] it might even have a detrimental effect on layer integrity compared with surfaces with no oxygen-inhibition layer.[35] Nevertheless, other studies suggest that an inhibited surface layer is required to increase bond strengths between resin composite increments.[36,37] For curing systems sensitive to oxygen, it might be reasonable to expect that a resin-rich surface would provide improved integrity between the incremental layers compared with a
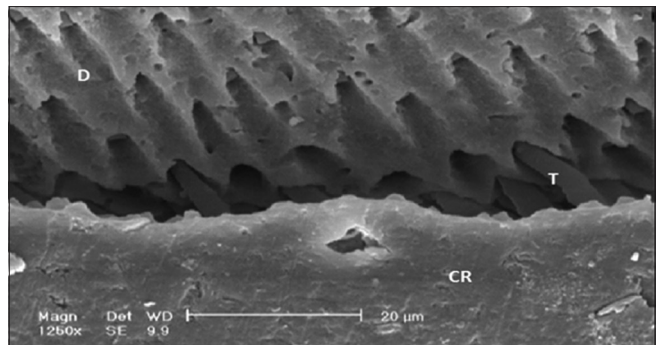
Figure 3: Standard error mean image of interface of (OptiBond Solo Plus + LS Bond) and dentin (×1250). CR: Composite resin; T: Resin tag; D: Dentin.
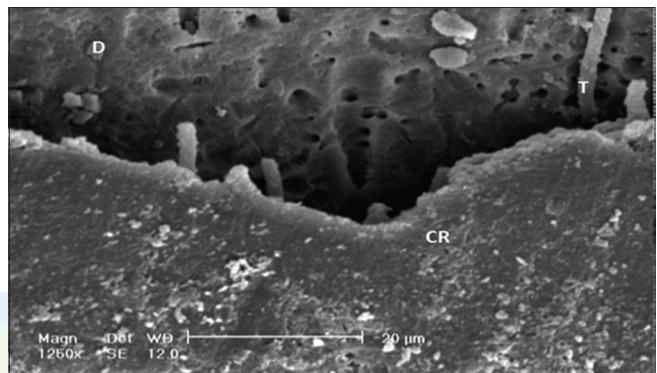
Figure 4: Scanning electron microscopy image of the interface of (Clearfil SE-Bond + LS Bond) and dentin (×1250). CR: Composite resin; T: Resin tag; D: Dentin.
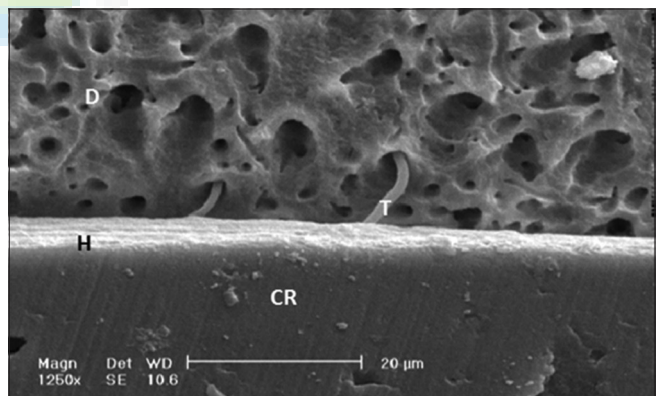
Figure 5: Scanning electron microscopy image of the interface of Silorane System Adhesive and dentin (×1250). CR: Composite resin; T: Resin tag; D: Dentin; H: Hybrid layer.

surface with no OIL since chemical bonds within interpenetrating networks would form more readily.

The ring-opening reaction of the silorane is a cationic polymerization reaction where no oxygen inhibition exists on the polymerized surface. Therefore, the bond between the successive layers depends only on the

chemical reactivity of the composite material.[4] It was hypothesized that the bond strength between the two successive layers of silorane would be lower than a methacrylate-based composite resin. Tezvergil *et al.*[20] evaluated shear bond strength of methacrylate-based composite resin (MBC) to silorane-based composite resin (SBC) with and without the use of phosphorylated methacrylate resin (SSA).In their study, MBC–MBC combination showed the highest mean shear bond strength values with no adhesive failures, followed by SBC–SBC, SBC –silorane-based intermediate resin – MBC, SBC –methacrylate-based intermediate resin–MBC and SBC–MBC.

Silorane adhesive resins are based on methacrylate chemistry with phosphate groups. Reaction of oxirane with phosphate groups and dimethacrylate with acrylate groups might be the reason for increased bond strength obtained.

Another reason may be the higher viscosity of phosphate–methacrylate silorane resin compared to dimethacrylate-based resin that provides a thick layer, which would act as an elastic layer; moreover, this layer could partly compensate the shrinkage stress developed by the free radical polymerization of dimethacrylate-based composite resin. In their study, it was shown that in order to bond MBC to SBC, a phosphate methacrylate-based intermediate resin (SSA) is required. In the present study, MBC provided adequate bond strength between SBC and dentin even without the use of LS Bond. In their study, application of a methacrylate-based intermediate layers led to 91.7% of adhesive failures while the use of silorane-based intermediate layers resulted in cohesive failures. However, in the present study, adhesive fractures were the most frequent type of fractures and cohesive fractures in compositeresins were the least frequent types, but this difference was not statistically significant.

Ivanovas *et al.*[21] reported that the highest shear bond strengths for repairing SBC with MBC were obtained when silorane was used with a dimethacrylate-based adhesive resin. Since the group, in whicha methacrylate-based adhesive was used only between SBC and MBC, showed a low bond strength value, it is hypothesized that the silane is the decisive factor for adhesion between the silorane surface and low-viscosity resin. In that study, a higher bond strength was obtained when a silorane-based flowable material was used as an intermediate agent for the silorane–silorane repair combination compared to using Silorane System Adhesive. It should be noted that SSA is not silorane-based

but it is phosphatedimethacrylate-based. The effect of using siloraneflowable for silorane repair might be the result of its stress-bearing ability due to higher elasticity and more stable chemical reaction of SBC and silorane-based flowable resin than the bond between the phosphate groups of the phosphatedimethacrylate-based adhesive (SSA) and SBC. In addition, when they applied only Heliobond (a methacrylate-based flowable resin) between SBC and MBC, adequate bond strength was obtained. Similar to these findings, we showed in this study that applying methacrylate-based adhesive between SBC and dentin resulted in sufficient bond strength.

Zheng *et al.*[38] showed that bond strength values significantly improved when the thickness of the adhesive layer increased. The authors supposed that thicker adhesive layer may permit "self-alignment" of the specimen during tensile testing, help stress distributions and result in higher bond strength values. The elastic behavior of dentin–composite interfaces can be affected by the use of filled or unfilled adhesive resins.[39,40] In order to increase bonding layer thickness, inorganic fillers have been added to dentin bonding systems that also increase their viscosity.[41]

Coelho *et al.*[42] reported that microtensile bond strength of filled adhesives is higher than unfilled adhesives. In our study, micro-shear bond strength of two adhesive systems (OptiBondSolo Plus, SiloraneSystem Adhesive) was higher than Clearfil SE Bond. This might be related to higher viscosity of Opti Bond Solo Plus and Silorane System Adhesive and more thickness of their adhesive layers. Another possible reason for these results may be the interaction of their phosphate groups of SE Bond with phosphate groups of silorane-based composite resin. However, further studies are needed to confirm this hypothesis.

CONCLUSION

Based on the results of this *in vitro* study, application of total-etch and self-etch methacrylate-based adhesives with and without use of a hydrophobic resin coating results in acceptable bond strengths.

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Conflicts of interest
The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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