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ORIGINAL ARTICLE

Effect of surface treatment on the initial bond strength of different luting cements to zirconium oxide ceramic

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Abstract The objective of this study was to compare the shear bond strength to zirconium oxide ceramic of adhesive-phosphate-monomer-containing (APM) and non-APM-containing (nAPM) luting cements after different surface treatments. nAPM cements: Bifix QM, Dual Cement, Duo Cement Plus, Multilink Automix, ParaCem Universal DC, PermaCem Smartmix, RelyX ARC, Variolink Ultra, and Variolink II; APM cements: Panavia EX, Panavia F2.0, and RelyX UniCem. Groups of ten test specimens were each prepared by layering luting cement, using cylindrical Teflon molds, onto differently treated zirconium dioxide discs. The surface treatments were airborne-particle abrasion with 110 μm alumina particles, silica coating (SC) using 30 μm alumina particles modified by silica (Rocatec System) or SC and silanization. Bifix QM and Multilink Automix were used in combination with an additional bonding/priming agent recommended by the manufacturers. After 48 h of water storage, each specimen was subjected to a shear test. Combinations involving APM-containing cements (14.41–23.88 MPa) generally exhibited higher shear bond strength than those without APM (4.29–17.34 MPa). Exceptions were Bifix QM (14.20–25.11 MPa) and Multilink Automix (19.14–23.09 MPa) in combination with system-specific silane or priming agent, which were on the upper end of shear bond strength values. With the use of the Rocatec system, a partially significant increase in shear bond strength could be achieved in nAPM cement. Modified surface treatment modalities increased the bond strength to zirconium oxide, although the most important factor in achieving a strong bond was the selection of a suitable cement. System-specific priming or bonding agents lead to further improvement.

Keywords Bond strength · Luting cements · Zirconium oxide · Surface treatment · Ceramic · Zirconia

Introduction

Since the first appearance of zirconium dioxide as a dental material, its range of application has continually expanded [28]. Although initially used for endodontic dowels and implant abutments [8, 12], subsequent in vitro studies delivered promising results for these indications [20, 40] and extended its use to single crowns [27, 35] and posterior three-unit fixed partial dentures, where the superior mechanical properties, compared to other ceramic materials like lithium disilicate- or alumina-based systems, are especially advantageous [31]. The clinical performance of zirconia post-and-cores and implant abutments in the anterior region were proven to be effective in long-term studies [10, 21, 25]. Vult von Steyern et al. [33, 34] published results from 2- and 3-year clinical studies on all-ceramic fixed dental prostheses designed according to the DC-Zirkon® technique. In these investigations, the success rates were 100% for three- to five-unit FDPs. Similar findings were reported by Sailer et al. [30] after a 5-year follow-up of posterior FDPs processed with a prototype CAM-system.

Some authors postulate that the advantages of adhesive fixation of all-ceramic crowns and bridges are due to

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improved retention, better marginal adaptation, and fracture resistance [2]. Luting cements showed higher bond strength to dental hard tissues than glass ionomer cement [1]. However, conventional cementation of zirconia restorations as recommended by some manufacturers seems to be sufficient due to the high fracture resistance of the material and retention values similar to adhesive luting procedures [9, 24]. One focus of current ceramics research is the development of materials and treatment protocols to expand the range of indication. Such an indication could be found in minimal invasive adhesive FDPs [38]. While in vitro investigations indicate sufficient fracture strength [15], reliable clinical data on the longevity of bonding to zirconia surfaces is lacking.

Although hydrofluoric acid etching and the application of a silane coupling agent to silica-based ceramics increases the bond strength between all-ceramic restorations and composite resins [4, 23], these techniques do not improve the bond strength of zirconium and alumina ceramics, as their high crystalline content renders them resistant to acid etching [7, 43]. Studies of shear bond strength to zirconia ceramic have shown that a composite resin cement containing an adhesive phosphate monomer (APM) provides significant bond strength values [3, 14]. The application of a phosphate monomer containing bonding/ silane-coupling agent mixture alone [3] or in combination with tribochemical silica coating (SC) seems to further increase the shear bond strength between zirconium oxide ceramic and a resin luting agent [2].

The purpose of this study was to evaluate the initial shear bond strength to planar zirconia surfaces coated with various luting agents (APM-containing and non-APM-containing) after different surface treatments [airborne-particle abrasion (APA) of alumina particles, SC, or SC and silanization (SCS)].

The working hypothesis was as follows: Independent from different surface treatments, APM-containing luting agents provide higher shear bond strength to zirconium oxide ceramic compared to non-APM-containing luting agents.

Materials and methods

Zirconium oxide rods (Digizon-A, AmannGirrbach, Pforzheim, Germany), 10 mm in diameter, were embedded in methacrylate (Palapress Vario, HeraeusKulzer, Wehrheim, Germany) and sliced using a diamond saw (Seccutom 10, Struers, Willich, Germany) with coolant at 1,700 rpm and a crosshead speed of 0.080 mm/s. The resulting sections had a thickness of 3 mm. The surfaces of the zirconium oxide discs were ground and polished with a diamond grinding disc and coolant (grain size, 40 µm) in a preparation system (Tegra Force 5, Struers, Willich, Germany). Finally, the specimens were cleaned in 96% ethanol for 5 min.

The 12 tested luting cements are presented in Table 1. Three of these cements contain phosphoric acid monomers: Panavia EX (Kuraray, Osaka, Japan), Panavia F 2.0 (Kuraray), and Rely X UniCem (3M Espe, Seefeld, Germany).

All shear test specimens (N=360) were prepared and divided into 12 groups (N=30). All specimens were corund-blasted with 110 µm alumina particles (pressure, 2.8 bars; application time, 10 s; 10 mm working distance), and the following three different surface treatment protocols were performed on ten specimens within every cement group: no further treatment (APA) and silica coating (SC) using 30 µm alumina particles (2.8 bars, 13 s, 10 mm) modified by silica or SC and SCS with Espe Sil (3M Espe). All surface processing was performed according to the Rocatec protocol (3M Espe).

After preparing the zirconia discs, Teflon molds having an inner diameter and height of 3 mm each were positioned in the center of the specimens and filled with the luting cements. The bonding procedures followed the manufacturers' recommendations. Light was applied to the top of the filled molds for 40 s (Elipar FreeLight, 3M Espe). After careful removal of the Teflon molds, every side of the cement cylinder was light-cured for a total of 80 s. The groups Bifix QM APA and Bifix QM SC had a system-specific silane (Keramik Bond, Voco) applied prior to layering the composite cylinder. Multilink Automix was used together with a specific primer containing phosphonic acrylate (Metal/Zirconia primer, Ivoclar Vivadent) recommended from the manufacturer on the APA and SC surfaces.

Prior to testing, all bonded specimens were stored in water at 37°C for 48 h [17].

Finally, shear bond strength was tested with a universal testing machine (ProLine Z005, Zwick, Ulm, Germany) at a crosshead speed of 0.5 mm/min using a jig according to ISO/TR 11405 (Guidance on testing of adhesion to tooth structure). Loads were converted automatically to MPa by dividing the failure load (Newtons) by the bonding area (square millimeter).

The data were analyzed by the Kruskall–Wallis test followed by pairwise comparisons using the non-parametric Mann–Whitney U test (α=0.01).

Specimen surfaces were examined with a light microscope (M5-48436, Wild, Heerbrugg, Switzerland) at ×12 original magnification to assess the mode of failure (adhesive at the interface between zirconium oxide surface and resin cement or cohesive within the resin cement).

Results

The usual parametric analysis of variance model analysis was not supported because the distribution of measured

Table 1 List of tested luting cements

System	Manufacturer	Components	Main composition[a]
Bifix QM	VOCO, Cuxhafen, Germany	Base, catalyst	*bis*-GMA, DMA, silica fillers, initiator, pigment, additives
Dual Cement	Ivoclar, Ellwangen, Germany	Base, catalyst	UDMA, DMA, SiO_2-fillers sil., ytterbium fluoride, catalysts, stabilizer, pigment
Duo Cement Plus	Coltene/Whaledent AG, Altstätten, Switzerland	Base, catalyst	Bis-GMA, Bis-EMA, TEGDMA, ba-glass sil., silica
Multilink Automix	Ivoclar, Ellwangen, Germany	Pastes A and B	DMA, HEMA, ba-glass fillers, ytterbium fluoride, SiO_2-fillers, catalysts, stabilizer, pigment
		Primer	Phosphonic acid acrylate, solvent, HEMA, Bis-EMA, stabilizer, initiator
Panavia EX	Kuraray, Osaka, Japan	Powder, liquid	BPEDMA, MDP, DMA, silica, barium sulfate, titanium oxide, DBP
Panavia F2.0	Kuraray, Osaka, Japan	Paste A	α-Quartz powder sil., ba-glass powder sil., titanium dioxide sil., MDP, sodium fluoride, 2,2-*bis*[methacryloxy(poly)ethoxyphenyl] propane, 1,2-*bis*(3-methacryloxy-2-hydroxypropoxy) ethane, neopenthylglycol-dimethylacrylate, 2,4, 6-trimethylbenzoyl-diphenyl-phosphinoxide, triethanol amine
		Paste B	*N*,*N*-Diethanol-*p*-toluidine, inhibitor, pigment
ParaCem Universal DC	Coltene/Whaledent AG, Altstätten, Switzerland	Base, catalyst	MA, BP, silica fillers
PermaCem Smartmix	DMG, Hamburg, Germany	Base, catalyst	Bis-GMA, activator, catalysts, additives
RelyX UniCem	3M ESPE, Seefeld, Germany	Powder	Glass powder, initiator, silica sil., pyrimidine, calcium hydroxide, peroxy compound, pigment
		Liquid	Methacrylated phosphoric ester, DMA, acetate, stabilizer, initiator
RelyX ARC	3M ESPE, Seefeld, Germany	Base, catalyst	*bis*-GMA, TEGDMA, zirconia filler, silica filler, dimethacrylate-polymer functionalized, amine, initiator, peroxide, BP, pigment
Variolink Ultra	Ivoclar, Ellwangen, Germany	Base, catalyst	UDMA, *bis*-GMA, barium glass sil., ytterbium fluoride, TEGDMA, spheric mixed oxide sil., catalysts, stabilizer, pigment
Variolink II	Ivoclar, Ellwangen, Germany	Base, catalyst	Bis-GMA, UDMA, TEGDMA, barium glass sil. ytterbium fluoride, mixed oxide sil, Ba-Al-fluoro-silicate glass, catalysts, stabilizer, pigment

[a] According to the information provided by the manufacturers.

bis-GMA bisphenol-A-polyethoxy dimethacrylate, *sil.* silanized, *DMA* dimethacrylates, *UDMA* urethane dimethacrylate, *bis-EMA* ethoxylated bisphenol A glycol dimethacrylate, *TEGDMA* triethylene glycol dimethacrylate, *HEMA* 2-hydroxy ethyl methacrylate, *ba* barium, *BPEDMA* bisphenol-A polyethoxydimethacrylate, *MDP* 10-methacryloyloxy-decyl dihydrogenphosphate, *DBP* dibenzoyl peroxide, *MA* methacrylates, *BP* Benzoyl peroxide, *Al* aluminum

shear bond strength values was non-normal. Non-parametric analyses were performed using the Kruskal–Wallis test, and for pairwise comparisons, the Mann–Whitney U test was used. Moderate correction for multiple testing was achieved using a level of α=0.01, instead of the customary α=0.05.

The observed differences of mean shear bond strength values between the tested groups depended on surface treatment and luting agent, indicating severe interactions among these variables. Table 2 presents a summary of the pairwise comparisons among the rank-ordered mean shear bond values. Treatment combinations with continuous vertical symbols were not statistically different. Rankings are represented from highest (top) to lowest (bottom) shear bond strength. As can be seen, there is an evident significant overlap of groups.

Overall, the highest value for mean bond strength was observed when Bifix QM was applied on a silica-coated (SC) surface. There were no significant differences compared to this value when RelyXUnicem/APA, RelyXUnicem/SCS, Multilink Automix/SC Panavia Ex/APA, Multilink Automix/SCS, RelyXUniCem/SC, Panavia F 2.0/APA, and Panavia EX/SCS were tested (Table 2).

As a result of the SCS treatment of the zirconium dioxide surface, the bond strengths of Dual Cement,

Table 2 Comparisons among rank-ordered shear bond strengths (mean in MPa) using Mann–Whitney *U* test at α=0.01

	ST	Mean (MPa)	SD (MPa)	Groupings
Bifix QM	SC	25.11	4.86	O
Rely X Unicem	APA	23.88	3.39	O +
Rely X Unicem	SCS	23.14	2.11	O + O
Multilink Automix	SC	23.09	6.41	O + O +
Panavia EX	APA	21.49	3.14	O + O +
Multilink Automix	SCS	21.18	7.41	O + O + O
RelyXUniCem	SC	20.23	3.66	O + O + O
Panavia F2.0	APA	19.33	3.75	O + O + O
Panavia EX	SCS	19.30	2.62	O + O + O
Multilink Automix	APA	19.14	4.32	+ O + O +
Panavia F2.0	SCS	18.60	2.62	O + O +
Variolink Ultra	SCS	17.34	4.05	+ O + O
Variolink II	SCS	16.76	6.40	+ O + O +
Panavia F2.0	SC	15.59	1.67	+ O + O +
Bifix QM	APA	15.56	2.62	+ O + O +
ParaCem Universal DC	SCS	14.98	5.99	+ O + O + O
Panavia EX	SC	14.41	1.76	O + O + O
Bifix QM	SCS	14.20	3.53	O + O + O
RelyXARC	SCS	12.35	5.28	+ O + O +
Dual Cement	SCS	12.17	3.21	O + O +
Duo Cement Plus	SCS	11.04	3.01	+ O +
RelyXARC	SC	11.03	3.94	+ O +
Variolink Ultra	SC	11.00	3.38	+ O +
Variolink Ultra	APA	9.68	2.73	O + O
RelyXARC	APA	9.54	2.82	O + O
PermaCem Smartmix	SCS	9.28	1.93	O + O
PermaCem Smartmix	SC	8.91	0.72	O + O
Variolink II	SC	8.34	1.49	+ O +
ParaCem Universal DC	SC	7.50	2.83	O + O
Variolink II	APA	6.81	2.18	+ O
PermaCem Smartmix	APA	6.46	1.25	O +
Duo Cement Plus	APA	5.69	3.92	+
Duo Cement Plus	SC	5.57	0.97	+
ParaCem Universal DC	APA	5.56	1.49	+
Dual Cement	SC	4.98	0.51	+
Dual Cement	APA	4.29	0.68	+

ST surface treatment

[a] Combinations joined with continuous vertical symbols (+ or O) are not statistically different

ParaCem Universal DC, PermaCem, Variolink II, and Variolink Ultra were significantly improved compared to APA and SC (Fig. 1).

Variolink Ultra SCS showed the highest bond strength of the non-APM cements without the addition of a system-specific primer or silane coupling agent.

Of the APM-containing cements, RelyXUnicem/APA resulted in the strongest bond, and Panavia EX/SC exhibited the weakest bond (Fig. 2).

Combinations involving APM-containing cements generally had higher shear bond strengths than those without APM. Exceptions were Bifix QM and Multilink Automix in combination with system specific silane or priming agents, which were on the upper end of the measured shear bond strength values. Specimens with non-APM-containing cements and specimens pretreated with APA or SC were near the lower end, while pretreatment with SCS was in the middle region of the observed bond strength spectrum.

All fractures occurred between the ceramic surface and the cements (adhesive mode).

Discussion

After studying the current literature, it is obvious that the dental profession has not agreed on a universally accepted bond strength test for resin composites bonded to ceramic. Tensile, flexural, and shear tests have been used to measure the resin-ceramic bond strength [5, 19]. Della Bona and van Noort [6] stated that a notable feature of publications on the bond strength of resin to ceramics is the observation that the failure mode is often cohesive within the ceramic base rather than at the adhesive interface. They concluded that the bond strength exceeds the cohesive strength of the ceramic. Based on this study, it appears as though shear bond strength data in the cylinder-on-disc experimental design are governed by the cohesive strength of the base material and does not actually describe the quality of the adhesive bond. Later, based on FEA observations of a highly non-uniform stress distribution in the adhesive interface, Della Bona and van Noort [6] postulated that the shear test can lead to false interpretations of bond strength values. As a result, the tensile bond strength test was suggested to be a more appropriate test arrangement for evaluating the bond strength of resin composite to ceramic.

However, it was decided to measure bond strength with the shear test in the present investigation. Cohesive fractures within the zirconia ceramic base were not expected to occur, as they had never before been reported. Moreover, when searching the current literature on bonding to zirconia, tensile bond strength tests can be found [16], especially from the scientific group around Kern [14, 37, 39], although the majority of publications refer to shear bond strength values [11, 22, 23, 26, 29, 32, 42]. The chosen test design promised to provide a good comparability of the obtained results with previously published data.

The material and fabrication of test cylinders may have an influence on shear test results. Again, a large variety of possible interpretations of a standard test could be found. While some authors used metal cylinders due to better resistance to pretreatment procedures as well as better detection and removal of excess cement [17], most investigations used composite cylinders [2, 3]. Few publications reported on bonding zirconia to zirconia using discs of different diameters [32, 42]. We decided to mold

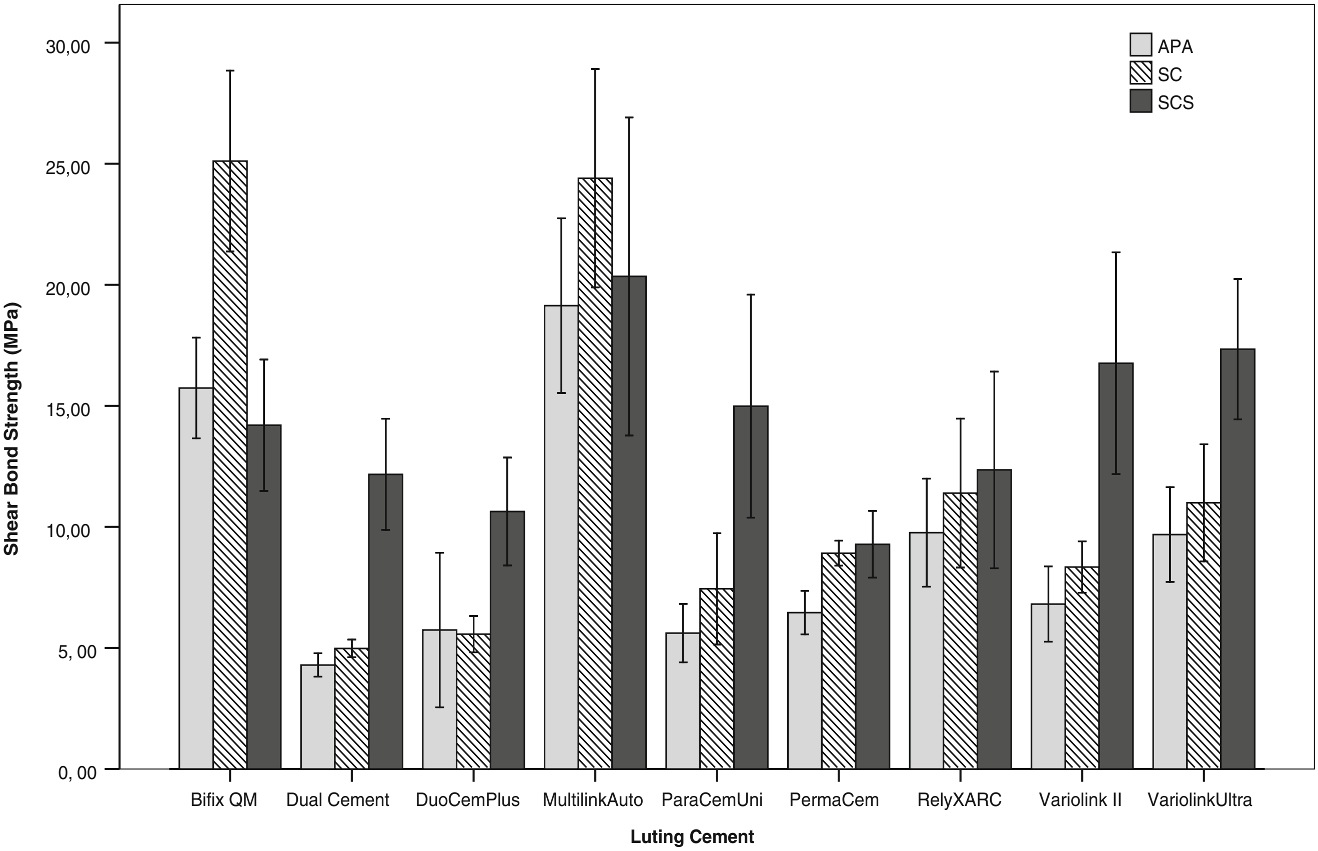

Fig. 1 Bond strength of non-APM-containing luting cements to ZrO_2 surfaces

the complete cylinder from luting cement by layering the material directly on the conditioned ceramic base. This procedure was also applied from Matinlinna et al. [18] and avoided any excess cement, which was considered to be important.

Bifix QM SC showed the highest values for initial shear bond strength in this study. This product was applied in combination with a system specific silane-coupling agent according to the manufacturer's recommendations. This silane-coupling agent seems to work more efficiently than the Rocatec-specific Espe Sil.

Similarly, strong bonding was achieved with Multilink Automix on a silica-coated zirconia surface. The manufacturer recommends the use of Zirconia primer (Ivoclar Vivadent), which contains APM. Atsu et al. [2] showed that the application of an MDP containing bonding/silane-coupling agent mixture increased the shear bond strength between zirconium oxide ceramic and resin cement. They also gave the explanation that a better wettability was obtained. The improved values found in our own investigation with SC compared with APA may be due to an increased surface area resulting from the additional treatment with 30 μm silicon dioxide particles.

Nevertheless, with the exception of Bifix QM and Multilink Automix, non-APM containing cements showed lower values than APM-containing cements in general, which is consistent with previous studies [3, 14, 37] and demonstrates that resin luting cements containing APM can bond strongly to sandblasted zirconia. Reactions may be formed between hydroxyl groups in the adhesive phosphate monomer and hydroxyl groups on the zirconia ceramic surface, similar to the chemical effects reported on the bond of phosphate ester monomer to metal oxides such as chromium, nickel, and aluminum [41, 42]. Comparing the surface treatments APA and SCS in the APM groups, no

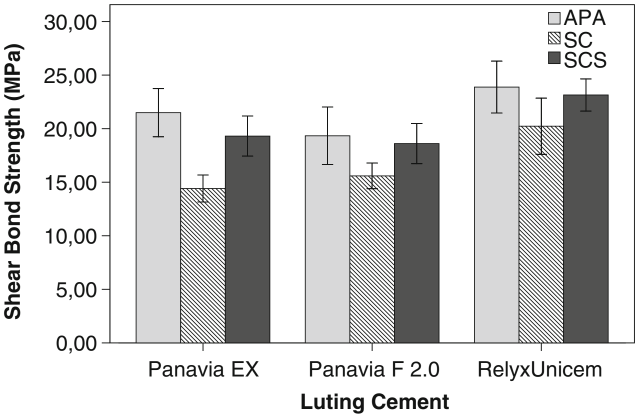

Fig. 2 Bond strength of APM-containing luting cements to ZrO_2 surfaces

significant differences could be found. This suggests that there may be no requirement for extensive pretreatment procedures as represented by the Rocatec system.

In general, SC and SCS resulted in a mostly significant increase of bond strength values for nAPM-based cements compared to airborne particle abrasion with alumina only. Similar results were presented by Kim et al. [16]. During the SC process, alumina particles modified with silica acid are blasted, and high heat is produced, which, together with pressure, cause the silica acid-modified alumina particles to be embedded within the ceramic surface. This is a prerequisite for an effective reaction of silane agents with zirconia ceramic [13].

In general, the working hypothesis of the present study can be accepted; an exception is the nAPM-containing Bifix QM when applied in combination with system specific silane.

Here, a large number of currently available resin luting cements in combination with various conditioning methods was tested, and previous conclusions regarding adhesive bonding on zirconium oxide were confirmed. The expanding applications of zirconium oxide ceramics in restorative dentistry, especially as framework material for adhesive FDPs, necessitates further research on luting procedures. APM-containing luting agents delivered promising results. However, conclusions from this study are limited, as no aging simulation such as longtime water storage or thermal cycling took place, which may significantly affect the results [36]. Furthermore, randomized clinical trials are needed to give reliable recommendations to the dental practitioner.

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Conflict of interest statement The authors declare that they have no conflict of interest.

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