EVALUATION OF THE SHEAR BOND STRENGTH OF BIODENTINE, PRE-MIXED NEOPUTTY AND NEW RESIN MODIFIED CALCIUM SILICA CEMENT WITH BULK FILL COMPOSITES; SCANNING ELECTRON MICROSCOPY-ENERGY DISTRIBUTED X-RAY SPECTROSCOPY ANALYSIS

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Abstract

The aim of this study was to evaluate the bond strength of three different calcium silicate based cements (Biodentine, NeoPutty and MTA Cem LC) with two different bulk fill composites (fiber-reinforced and non-fiber-reinforced). Plexiglas molds with a diameter of 4 mm and a thickness of 2 mm were prepared (n=60). Each CBC was mixed according to the manufacturer's instructions and placed in plexiglass molds. The BD, NP, and MTA Cem LC samples were then randomly divided into 2 subgroups containing 10 samples each. The surfaces of the samples were air dried and Single Bond universal adhesive was applied. Then, cylindrical plastic capsules of 2 mm height and 2 mm inner diameter belonging to Filtek Bulk fill and EverX Posterior composite resin groups were centered on the coating material and polymerized for 20 seconds. After SBS testing, all samples were examined by scanning electron microscopy (SEM) to identify failure patterns. Three samples, one from each group, were prepared to evaluate the chemical compositions of the materials. Samples were prepared using plexiglass molds with a diameter of 10 mm and a thickness of 2 mm. The materials were then examined with an energy dispersive X-ray spectroscopy for surface elemental analysis. The values obtained from the tests were evaluated as statistically significant ($p < 0.05$). After SBS testing, the difference between all CBCs was statistically significant in both compound groups. According to the findings obtained from this study, it was concluded that MTA Cem LC had the highest SBS values in both composite groups.

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ABSTRACT

The aim of this study was to evaluate the bond strength of three different calcium silicate based cements (Biodentine, NeoPutty and MTA Cem LC) with two different bulk fill composites (fiber-reinforced and non-fiber-reinforced). Plexiglas molds with a diameter of 4 mm and a thickness of 2 mm were prepared (n=60). Each CBC was mixed according to the manufacturer's instructions and placed in plexiglass molds. The BD, NP, and MTA Cem LC samples were then randomly divided into 2 subgroups containing 10 samples each. The surfaces of the samples were air dried and Single Bond universal adhesive was applied. Then, cylindrical plastic capsules of 2 mm height and 2 mm inner diameter belonging to Filtek Bulk fill and EverX Posterior composite resin groups were centered on the coating material and polymerized for 20 seconds. After SBS testing, all samples were examined by scanning electron microscopy (SEM) to identify failure patterns. Three samples, one from each group, were prepared to evaluate the chemical compositions of the materials. Samples were prepared using plexiglass molds with a diameter of 10 mm and a thickness of 2 mm. The materials were then examined with an energy dispersive X-ray spectroscopy for surface elemental analysis. The values obtained from the tests were evaluated as statistically significant ($p < 0.05$). After SBS testing, the difference between all CBCs was statistically significant in both compound groups. According to the findings obtained from this study, it was concluded that MTA Cem LC had the highest SBS values in both composite groups.

Key Words: NeoPutty, Fiber-Reinforced Bulk Fill Composite, Shear Bond Strength, SEM

Research Highlights

The results of this study showed that the light-cured calcium silicate cement has a higher bond strength value than conventional calcium silicate-based cements. Clinically, this is important to the success of restorations.

INTRODUCTION

Calcium silicate based bioactive cements (CBCs) are used in treatments such as pulp capping, pulpotomy, apexogenesis, apexification, perforation and resorption repair, and apical surgery. (İpek et al., 2022; Maden & Altun, 2013; Taha et al., 2017). Mineral trioxide aggregate (MTA) was the first calcium silicate-containing biomaterial used in dentistry (MTA) (Jefferies, 2014). This material has the advantages of stimulating hard tissue formation, antibacterial activity, releasing calcium hydroxide, low solubility, high impermeability and setting in a wet environment (Altunsoy et al., 2015; Asgary et al., 2007; Camilleri et al., 2014). Despite all these advantages, it has several disadvantages such as long setting times, manipulation difficulties, high costs and causing discoloration on teeth. In order to eliminate these disadvantages and to improve the physicochemical and mechanical properties of the material, alternative new materials have been tried to be produced by the manufacturer (Nikhade et al., 2016; Srinivasan et al., 2009). Pre-mixed tricalcium silicate-based cements have been introduced to overcome the potential disadvantage of the heterogeneous consistency that occurs when mixing conventional tricalcium silicate-based cements with powder and liquid (Loushine et al., 2011; Persson & Engqvist, 2011). Pre-mixed hydraulic cements are exposed to atmospheric humidity on removal from their containers and therefore generally have a short shelf life after the can is opened (Guo et al.,

2016; Persson & Engqvist, 2011). NeoPutty (NP) (NuSmile, Houston, TX, USA) is a pre-mixed, ready-to-use bioactive tricalcium silicate-based material consisting of an extremely fine, inorganic tricalcium/dicalcium silicate powder that the manufacturer claims to overcome this problem (Sun et al., 2021).

Biodentine (BD) advantages such as shorter setting time and better mechanical properties enable it to be used as a permanent dentin replacement material (Kaur et al., 2017; Malkondu et al., 2014). The short setting time of this material allows the restoration to be done in a single visit (Aksoy & Ünal, 2017; Grech et al., 2013). It also has positive effects on vital pulp and stimulates tertiary dentin formation (Camilleri, 2013; Laurent et al., 2012). It shows similar properties with dentin in terms of modulus of elasticity and compression resistance. Compared to MTA, it has been reported to be more resistant to compression and bending forces (Bayram & Bayram, 2016; Kaur et al., 2017) less solubility, higher structural strength, and better capping (Bhat et al., 2014).

MTA Cem LC is a new light-cured, resin-modified, tri-calcium silicate-based material designed for use as a direct and indirect pulp capping as a protective base/primer under composites, amalgams, cements and other base materials.

To facilitate the placement of resin-based composites in large layers in the posterior region; Bulk fill composite resins (CR) that can be placed in a single layer or in thicker layers have been produced. Reinforcing composites with resin fibers is a preferred method today (Batmaz et al., 2021). Fiber-reinforced CRs consist of a combination of a resin matrix, randomly oriented E-glass fibers and inorganic particle fillers (Garoushi et al., 2018). Short fiber reinforced CR (EverX Posterior; GC, Tokyo, Japan) was introduced to mimic the stress-absorbing properties of dentin (Batmaz et al., 2021).

Various methods such as tensile, shear and push out tests are used to evaluate the bond strength of dental materials to dentin (Drummond et al., 1996; Huffman et al., 2009; Reyes-Carmona et al., 2010). Bonding strength tests can be evaluated qualitatively and quantitatively. While microscopic methods such as scanning electron microscopy (SEM) are used for qualitative evaluation of bonding, tests such as push-out, shear and tensile are used for quantitative evaluation (Goracci et al., 2007).

In this study, it was aimed to evaluate the bond strength of BD, NP and MTA Cem LC calcium silicate based cements to EverX Posterior and Filtek Bulk fill CR by shear bond strength test (SBS).

MATERIALS AND METHODS

In this study, 3 different CBCs and 2 different bulk fill CRs (fiber reinforced) were evaluated. The technical profiles of the materials used are shown in Table 1. Power analysis of the study was performed to determine the sample size, it was decided to take 10 samples from each group, and the power of the test was found as p = 0.9057.

Table 1. The technical profiles of materials used in the study

Material	Composition	Manufacturer	Method/steps for Application/ Setting Time
Biodentine	Powder: Tricalcium silicate, dicalcium silicate, calcium carbonate, zirconium oxide, calcium oxide, iron oxide Liquid: Calcium chloride as accelerator and water-soluble polymer.	Septodont, Saint Maur des Fosses, France	Mixing premeasured unit dose capsules in a high-speed amalgamator for 30 s. / 12 min

Material	Composition	Manufacturer	Method/steps for Application/ Setting Time
NeoPutty	Tricalcium silicate, dicalcium silicate powder, tantalum oxide (tantalite) in an anhydrous organic liquid	NuSmile, USA	4 hours
Mta Cem LC	BisGMA, CaO, silica, barium sulfate (BaSO4)	Nexobio, Korea	Light polymerize for 20 s.
EverX Posterior	Bis-GMA, EGDMA, 800μm length and 17μm diameter E-glass fibers, Barium glass fiber filler, Silicon dioxide	GC, Japan	Light polymerize for 20 s.
Filtek Bulk Fill	Bis-GMA, UDMA, BIS-EMA, zirconia/silica, ytterbium trifluorite filler	3M ESPE, USA	Light polymerize for 20 s.
Single Bond Universal	10-MDP, Dimethacrylate resins, HEMA, Methacrylate modified polyalkenoic acid copolymer, Ethanol, Water, Initiators, Silane	3M ESPE, Germany	Light polymerize for 20 s.

Preparation of Samples

Plexiglass molds with a diameter of 4 mm and a thickness of 2 mm were prepared (n=60). Each material was mixed according to the manufacturer's instructions and placed in plexiglass molds. After the materials were placed in the mold, mylar strips were placed on both surfaces. Excess material was removed by applying pressure with a glass plate. Before bonding, each sample was kept in a humidity chamber at 37°C and 100% humidity for 24 hours.

BD, NP and MTA Cem LC samples were then randomly divided into 2 subgroups of 10 samples each. The surfaces of the samples were air dried and Single Bond universal adhesive was applied according to the manufacturer's instructions. No acid etching was performed before the bonding system was applied in any of the study groups. Then, for Filtek Bulk fill and EverX Posterior composite resin groups, cylindrical plastic capsules of 2 mm height and 2 mm inner diameter were placed centrally on the coating material and polymerized for 20 seconds. a light emitting diode (LED) curing unit (Elipar Deepcure-L, 3M Espe, USA). All samples were stored at 37degC with 100% humidity for 24 hours before proceeding with the SBS test.

SBS Test

Each sample was placed in a universal testing machine ((Instron Lloyd LRX; Lloyd Instruments Ltd., Fareham, UK) and the cutting mode was selected. A chiseled plunger was mounted on the movable crosshead of the testing machine and positioned with its leading edge toward the base/adhesive interface. Next, a chisel-shaped bar applied shear force at a crosshead speed of 1 mm/min until bond failure. SBS values calculated

via the division between the peak failure force (N) and the cross-sectional area of the bonded interface (3.14 mm2) , expressed in megapascals (MPa) (1 MPa = 1 N/mm2) (Cantekin & Avci, 2014).

Failure and SEM–EDX Analysis

SEM (Tescan MIRA3 XMU, Brno, Czech Republic) was used to evaluate the fracture type of samples applied with the SBS test. Samples (Quorum Q150R ES, Quorum Technologies, UK) were gold plated before being evaluated by SEM. The surface of the sample was scanned and representative areas showing the types of refraction were photographed at x50, x250, x500 magnification with an acceleration voltage of 10 kV.

To evaluate the chemical composition of CBCs, 3 samples were prepared, 1 sample from each group. Samples were prepared using plexiglass molds with a diameter of 4 mm and a thickness of 2 mm. Setting time was waited for the prepared materials in accordance with the manufacturer's instructions. The materials were examined with an energy dispersive X-ray spectroscopy (EDX) system for surface elemental analysis.

Data presentation and statistical analysis

Data were evaluated using the SPSS 22.0 (Statistical Package for Social Science Version: 22) program. Arithmetic mean and standard deviation are given in descriptive statistics. When the normality assumption was provided (Shapiro–Wilk), a one-way ANOVA analysis was performed. The Bonferroni test was used in paired comparisons. $p < 0.05$ was considered statistically significant.

RESULTS

According to the results of our study, the highest SBS value was observed in the Mta Cem LC group among the two composite groups. The lowest SBS value was observed in the NP group in both composite resin groups. In pairwise comparison, there was a statistically significant difference between all CBCs ($p < 0.05$). Although there was no statistically significant difference between the EverX Posterior CR and Filtek Bulk fill CR groups for all CBCs, higher bond strength was obtained in the EverX Posterior CR group. The results of the SBS were shown in Table 2.

Tablo 2. Mean shear bond strength values (MPa) and standard deviations (+-SD) of each group

	EverX Posterior CR	**Filtek Bulk Fill CR**
BD	12.23 ± 1.54	10.82 ± 1.55
NP	4.49 ± 2.23	3.17 ± 2.42
MTA Cem LC	25.19 ± 2.91	22.07 ± 1.73
p value	0.001*	0.001*

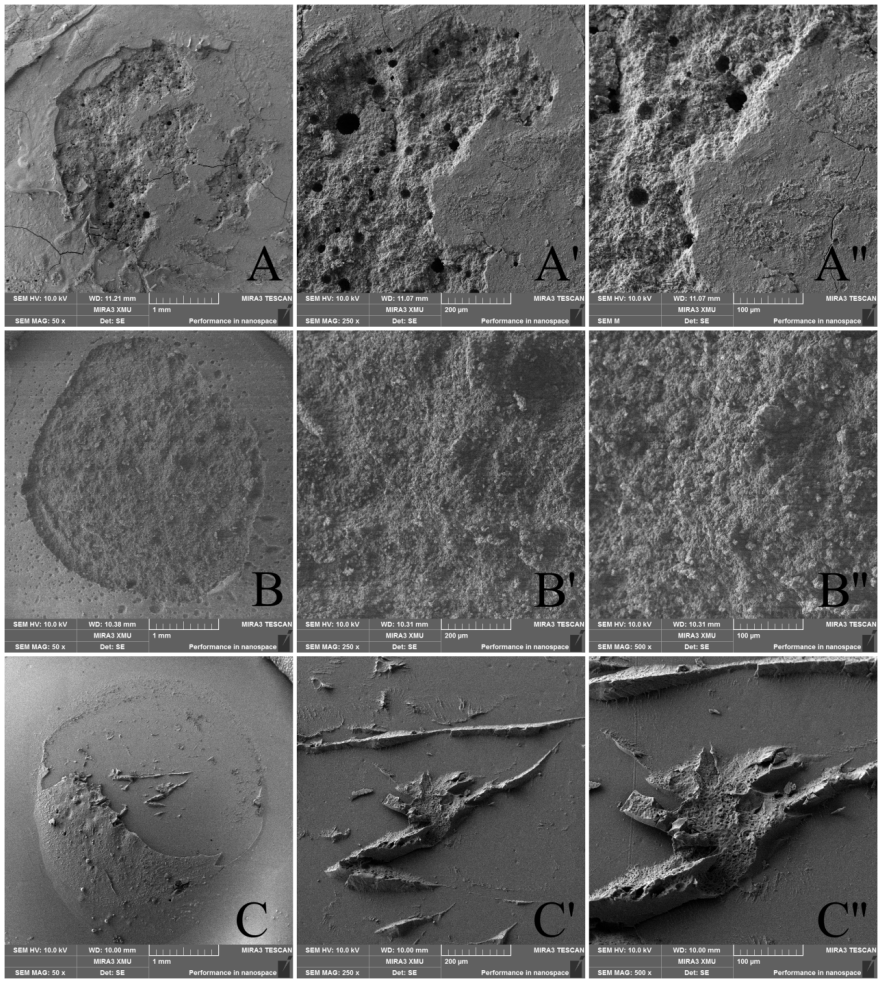
* *$p < .05$ was accepted as a significance level*

The surfaces of the materials after SBS test were evaluated by SEM and the images of the samples at x50, x250, x500 magnifications are shown in Figure 1. Cohesive and mixed failure types were predominantly observed for BD and MTA Cem LC, whereas adhesive failure type were predominant for NP. The results of the mode of failure analysis were shown in Table 3.

Table 3. Failure type of CBCs

	EverX Posterior CR	**EverX Posterior CR**	**EverX Posterior CR**	**Filtek Bulk Fill CR**
Mode of failure	Adhesive	Mixed	Cohesive	Adhesive
BD	2	4	4	1
NP	6	3	1	5
MTA Cem LC	2	5	3	1

Figure 1. Failure types after the push-out bond strength test A; cohesive failure in BD group, **B;** adhesive failure in NP group and C; mix failure in MTA Cem LC



All sample surfaces were gold (Au) coated for conductivity but Au was not included in EDX quantification. EDX analysis was performed to determine the elemental distribution of the materials and is shown in Table 4.

Table 4. SEM-EDX analysis results for CBCs

BD	**BD**	**NP**	**NP**	**MTA Cem LC**	**MTA Cem LC**
Element	**Weight %**	**Element**	**Weight %**	**Element**	**Weight %**
C	18.95	**C**	34.63	**C**	36.15
Ca	35.49	**Ca**	12.98	**Ca**	28.45
Si	10.78	**Si**	4.82	**Si**	8.63
O	32.47	**O**	20.89	**O**	22.96
Al	0.01	**Al**	2.92	**Al**	2.61
Zr	2.3	**Ba**	0.96	**Ba**	1.2
		Ta	22.8		
Total	100	**Total**	100	**Total**	100

DISCUSSION

CBCs are used self-curing hydraulic cements. CBCs are widely used in endodontic procedures involving pulpotomy, apexogenesis, apexification, perforation repair, root end filling, pulp regeneration, and hard tissue repair such as pulp capping. It has been reported that the widespread use of CBCs in these clinical treatments is due to their closure ability, tightness and biocompatibility. (Ma et al., 2011; Parirokh & Torabinejad, 2010; Sarkar et al., 2005; Wang et al., 2012).

The bond strength of CBCs to restorative materials is important for the quality and success of restorations. Because good bonding creates an adhesive bond that can spread stress relatively evenly over the bonding site (Oskoee et al., 2011). Under in vivo conditions, shear stresses are thought to be the cause of the deterioration of the adhesion of the materials and the formation of joint failures. Shear or tensile bond strength tests are the most commonly preferred test methods for in vitro evaluation of the performance of adhesive systems. For these reasons, the shear bond strength test was used in our study to predict the clinical performance of CBCs. (De Munck et al., 2005; Moll et al., 2004).

Many research has found that light-cured CBCs have higher SBS for composite structure or glass ionomer cement than conventional CBCs (Alhowaish et al., 2020; Alzraikat et al., 2016; Mehra et al., 2020; Samman et al., 2017). In our study, when the bond strength of the three CBCs we used was compared, a higher SBS value was obtained in MTA Cem LC in both composite groups. Deepa et al. (Deepa et al., 2016) in studies evaluating the bond strength of light-cured CBC and BD, a higher value was observed for light-cured CBC. Özata et al. (Özata et al., 2022) evaluating the bond strength of different CBCs, they reported that light-cured cement showed higher bond strength. This may be due to the resin content in MTA cem LC, which is a combination of a HEMA/TEGDMA based resin and calcium-silicate powder. It can be explained by the light activation of HEMA and TEGDMA monomers forming a polymeric network that can stabilize the outer surface of the cement. Thus, a chemical bond is formed with the composite resin. On the other hand, the absence of resin content in biodentine and neoputty suggests that the bonding of these materials to the resin composite is micromechanical (Ajami et al., 2013).

In our study, BD showed a lower bond strength value than MTA Cem LC but higher than NP in both bulk fill composite groups. The hydration process of calcium silicate cements goes through several stages, and the resulting hydrated cement crystallizes poorly in the early setting stages and is highly porous (Prati & Gandolfi, 2015). Adhesive agent application and cure shrinkage of the resin composite can strain the pores. At this early setting stage, immature BD adversely affects the bond strength of the cement. Therefore, at least 2 weeks are needed for BD to crystallize, to achieve sufficient bulk strength to withstand polymerization stresses (Hashem et al., 2014). Palma et al. (Palma et al., 2021) in their research where they evaluated the bond strength of BD immediately and after 2 weeks, they reported that it showed higher bond strength even after waiting for 1 week. In this study, attachment to BD was performed after 12 minutes to illustrate a single appointment clinical procedure. This may be the cause of the low bond strength and cohesion failures in BD. From studies evaluating the bond strength of BD, Krawczyk-Stuss et al. (Krawczyk-Stuss et al., 2019) found the bond strength of BD to be 9.39 MPa, and Odabaş et al. (Odabaş et al., 2013) found the bond strength of BD to be 9.127-11.057 MPa, Altunsoy et al. (Altunsoy et al., 2015) found 1.2 MPa, and Cantekin et al. (Cantekin & Avci, 2014) found 17.7 MPa. In our study, the bond strength of BD was found to be 10.82-12.23 MPa. These differences can be caused by differences in the adhesive systems (SE or TE) used, chemical content differences between bonding agents, the degree of polymerization or the depth of penetration in the composite or BD as a result of polymerization shrinkage at the interface. (Deepa et al., 2016; Ebrahimi et al., 2013).

NP, one of the premixed calcium silicate cements produced as an alternative to conventional powder-liquid cements, had the lowest SBS value in both composite groups in our study (3.17-4.49 MPa). Evaluating the bond strength of bioceramic materials, Alqahtani et al.(Alqahtani et al., 2022) found the lowest SBS value in the NP group (4.04 MPa), while Hursh et al. (Hursh et al., 2019) obtained the lowest value in the Endosequence putty group (4.47 MPa) in the premixed putty form. Palma et al.(Palma et al., 2021) evaluated the effect of restorative timing on the bond strength of calcium silicate-based cements, reported

that bioceramic cements in putty form showed low bond strength value when applied immediately. According to these findings, the low SBS of putty-formed CBCs may be related to the hydrophobic nature of the resin composites and the presence of acidic monomers in the universal adhesive system, causing erosion of the newly formed crystalline structure and the gel-like amorphous layer of the bioceramic material (Chang, 2012; Hidari et al., 2020).

Bulk fill CR provide advantages over conventional composites due to ease of application and low technical sensitivity. In addition, owing to its macromechanical properties, it acts as a dentin replacement in high stress areas. Shuwasih et al. (Chang, 2012) evaluated the bond strength of bulk fill and nano composites using SE adhesive system, bulk fill composites showed higher SBS value. Raina et al. (Raina et al., 2020) reported that bulk fill composites showed higher SBS values in their study where they evaluated the bond strength of CSC. Due to the high monomer concentration in conventional composites, the SBS value may be low as a result of polymerization shrinkage (Van Dijken & Pallesen, 2016). Although there are studies evaluating the bonding efficiency between bulk fill composite resins and conventional composite resins in the literature, no studies evaluating the bond strength of fiber-reinforced composite resins have been found. In our research, EverX Posterior (a fiber-reinforced bulk fill composite resin), showed higher bonding values for all CBCs compared to the 3m Filtek Bulk fill composite resin group. However, it was not statistically significant. In line with the limitations of our study, we think that the effect of fiber-reinforced bulk fill composites on bond strength is less.

While the interface bond between the adhesive and the adend is the adhesive failure, if the adhesive layer remains on both surfaces as a result of the failure, it is defined as cohesion failure (Ebnesajjad & Ebnesajjad, 2013). Therefore, the biomaterial in permanent restoration construction and SBS for permanent restoration and failure modes are also important for success in endodontics. In general, cohesive failure indicates that the materials have reached their maximum strength in adhesiveness. However, the failure mode is not the only criterion for measuring the success of adhesion (Ebnesajjad & Ebnesajjad, 2013; Tate et al., 1996). When the failures types were evaluated in our study, cohesive and mixed type failures were observed in the MTA CEM LC and BD groups, while adhesive and mixed type failures were observed in the NP group. The higher number of adhesive failures seen in the NP group may indicate that a strong chemical bond is not formed between the adhesive and the NP. However Özata et al. (Özata et al., 2022) reported that cohesive type failure was more prevalent in the NP group in their study. This difference can be explained by the difference in the adhesive used and the evaluation method.

Si and especially Ca have an osteoinductive effect in the formation of hard tissue such as dentin and bone. When the SEM-EDX results of our study were evaluated, Ca and Si were observed in three CSCs. Although there is alumina in MTA Cem LC and NP, very little amount was found in BD. It has been reported that alumina is responsible for tooth discoloration in the coronal use of MTA. (Felman & Parashos, 2013; Jang et al., 2013). However, since alumina is seen in calcium silicate-based cements produced later and does not cause discoloration, it is thought that it is not the main criterion for tooth discoloration (İpek et al., 2022). In addition, Ba which provides radiopacity, was found in trace amounts in MTA Cem LC and NP, but not in BD. However, there is zr oxide (Buła et al., 2020) in the BD to provide radiopacity. In addition, unlike other groups, tantalum oxide was observed in NP.

CONCLUSION

In all composite resin groups, MTA Cem LC showed better SBS than NP and BD. In MTA Cem LC and BD, cohesive and mix failure types were dominantly observed but NP adhesive failure type was dominantly.

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CONFLICT OF INTEREST

The authors deny any conflicts of interest related to this study.

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