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Bond strength of resin cements in semi-indirect restorations: an *in vitro* study

Resistência de união de cimentos resinosos em restaurações semi-indiretas: um estudo *in vitro*

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ABSTRACT

Objective: To evaluate in vitro the bond strength of different resin cements, both conventional and self-adhesive, used in semi-indirect restorations. **Material and Methods:** Bovine incisors (n = 48) were randomly divided into four groups (n = 12) according to the resin cement used for cementation: G1 - RelyX ARC (3M), G2 - RelyX U200 (3M), G3 - NX3 (Kerr), and G4 - Maxcem Elite (Kerr). Restorations were performed using the semi-indirect technique, simulated by molding the prepared cavities in the bovine incisors. Bond strength (MPa) was measured using a push-out mechanical test in a universal testing machine. The data were statistically analyzed using one-way ANOVA followed by Tukey's test ($\alpha = 0.05$). **Results:** Multiple comparison analyses demonstrated that the RelyX U200 (3M) group, a self-adhesive resin cement, exhibited the highest bond strength values (4.01 ± 1.28). A statistically significant difference was observed only in comparison with the Maxcem Elite (Kerr) group (2.73 ± 0.95) ($p \leq 0.015$). **Conclusion:** Cement formulation influenced bond strength. Although the self-adhesive resin cement RelyX U200 showed the highest values, another self-adhesive cement (Maxcem Elite) showed lower bond strength than the conventional cements (RelyX ARC and NX3), indicating variability within the same cement category.

KEYWORDS

Adhesiveness; Dental cements; Dental materials; Dental restoration, permanent; Resin cement.

RESUMO

Objetivo: Avaliar *in vitro* a resistência de união de diferentes cimentos resinosos, tanto convencionais quanto autoadesivos, utilizados em restaurações semi-indiretas. **Material e Métodos:** Incisivos bovinos (n = 48) foram aleatoriamente divididos em quatro grupos (n = 12) de acordo com o cimento resinoso empregado na cimentação: G1 – RelyX ARC (3M), G2 – RelyX U200 (3M), G3 – NX3 (Kerr) e G4 – Maxcem Elite (Kerr). As restaurações foram confeccionadas pela técnica semi-indireta, simulada por meio da moldagem das cavidades preparadas nos incisivos bovinos. A resistência de união (MPa) foi mensurada por meio do ensaio mecânico de *push-out* em máquina universal de ensaios. Os dados foram analisados estatisticamente por ANOVA de uma via, seguida do teste de Tukey ($\alpha = 0{,}05$). **Resultados:** As análises de comparações múltiplas demonstraram que o grupo RelyX U200 (3M), um cimento resinoso autoadesivo, apresentou os maiores valores de resistência de união (4.01 ± 1.28). Foi observada diferença estatisticamente significativa apenas em comparação com o grupo

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Maxcem Elite (Kerr) (2.73 ± 0.95) ($p \leq 0{,}015$). **Conclusão:** A formulação do cimento influenciou a resistência de união. Embora o cimento resinoso autoadesivo RelyX U200 tenha apresentado os maiores valores, outro cimento autoadesivo (Maxcem Elite) apresentou menor resistência de união em comparação aos cimentos convencionais (RelyX ARC e NX3), indicando variabilidade dentro da mesma categoria de cimento.

PALAVRAS-CHAVE

Adesividade; Cimentos dentários; Materiais dentários; Restauração dentária permanente; Cimentos de resina.

INTRODUCTION

Resin cements are widely used in clinical dental practice across various restorative procedures [1,2]. The organic matrix of these materials is primarily composed of methyl methacrylate, Bis-GMA dimethacrylate, or urethane dimethacrylate, combined with colloidal silica or barium glass fillers ranging from 20% to 80% by weight. These components form the base of the cement, providing flexibility and polymerization capacity [3]. The choice of a resin-based luting agent should be guided by consideration of its physical properties, mechanical characteristics, and handling technique [4]. Resin cements exhibit low solubility in the oral environment, lower susceptibility to marginal microleakage, and a wide variety of shades available for the clinician to choose from, compared to other luting agents [5].

Resin cements can be classified based on their mechanism of adhesion to dental structures, and can be conventional, requiring the prior application of an adhesive system, or self-adhesive, which do not require the separate application of an adhesive [6]. One of the main clinical indications for resin cement is semi-indirect restoration, a technique that combines the advantages of both direct and indirect approaches, offering the advantage of lower cost and the possibility of reconstructing extensive losses of tooth structure [7]. Polymerization is initiated on the model and further enhanced through heat curing, which improves tensile strength, elastic modulus, fracture resistance, hardness, and wear resistance [8].

Depending on the adhesion protocol and surface treatment, both conventional and self-adhesive resin cements can interact with the dentin substrate in different ways [9]. Clinical success in restorative procedures depends on multiple factors, the most critical being the bond strength between the resin cement, the dental substrate, and the restorative material [10]. Bond strength is a key aspect in the study of resin cements, as restorations, like natural teeth, are subject to compressive forces generated by occlusal loading [11]. In the literature, most of the available data on self-adhesive resin cements comes from studies on the cementation of posts in root dentin, which makes it necessary to understand how polymerization limitations in areas with limited light exposure, such as the apical third of the root canal, also apply to semi-indirect/indirect restorations. Modified self-adhesive systems can compensate for reduced light exposure and still provide clinically relevant adhesion [12].

Therefore, laboratory tests should aim to realistically simulate the forces and stresses present in the oral environment in order to generate results that reflect the clinical performance of the evaluated materials. In this context, the present study aimed to evaluate, through an in vitro assay, the bond strength of resin cements used in semi-indirect restorations. The null hypothesis tested was that there would be no significant difference in bond strength among the different types of resin cements used in semi-indirect restorations.

MATERIAL & METHODS

Study design

An in vitro laboratory study was conducted, in which the response variable was the bond strength. The experimental factors analyzed were the types of resin cement: RelyX ARC (3M Oral Care, St. Paul, MN, USA), RelyX U200 (3M Oral Care, St. Paul, MN, USA), NX3 (Kerr, Orange, CA, USA), and Maxcem Elite (Kerr, Orange, CA, USA). The composition of the materials is listed in Table I. The samples consisted of bovine teeth, prepared in a standardized manner and handled by two operators previously trained and instructed on the methodological protocol.

Table I - Materials and composition

Resin cement	Composition
Conventional RelyX ARC – 3M	BIS-GMA, TEGDMA, pigments, tertiary amine, benzoyl peroxide. Contain inorganic zirconia/silica particles with 67.5% by weight and average particle size of 1.5μm.
Self-adhesive RelyX U200 – 3M	Base paste: Silane-treated glass powder, 2-propenoic acid, 2-methyl, 1,1'-(1-(hydroxymethyl)-1,2-ethanodiyl) ester, triethylene dimethacrylate glycol (TEG-DMA), silane treated with silane, glass fiber, sodium and t-butyl per-3,5,5-trimethylhexanoate. Catalyst paste: Silane-treated glass powder, dimethacrylate substitute, silane treated silica, sodium p-toluenesulfonate, 1 benzyl5-phenyl-baric acid, calcium salts, 1,12-dodecane dimethacrylate, calcium hydroxide and titanium dioxide.
Conventional NX3 – Kerr	Uncured methacrylate ester monomers, HEMA,PTU,CHPO,free tertiary amines and benzoyl peroxide, inert mineral fillers, titanium dioxide, radiopaque agent, and pigments.
Self-adhesive Maxcem Elite – Kerr	HEMA,GDM,UDMA,1,1,3,3-tetramethylbutyl hydroperoxide TEGDMA, fluoroaluminosilicate glass, GPDM, barium glass filler, fumed silica (69 wt %).

Based on previous studies that evaluated displacement resistance using the push-out test, the appropriate sample size for a 5% level of statistical significance was estimated to range from 8 to 25 specimens per group [13-16]. Considering these parameters, 48 bovine incisors were selected and randomly assigned to four groups (n = 12), according to the resin cement used for cementation, as described in Table II.

Sample preparation

Bovine incisors free of cracks and structural defects were selected and disinfected in 0.1% thymol solution at 40°C for one week, then subjected to prophylaxis using pumice and water. The roots were removed using a model trimmer precisely at the cementoenamel junction (CEJ), and the crowns were sectioned with diamond discs using straight, transverse cuts to obtain disc-shaped samples measuring 4 mm in thickness, with a central void corresponding to the pulp chamber.

Cavity preparations were performed within the pulp chamber space using a standardization device equipped with a conical Maxicut bur, aiming to create standardized cavities (4 mm in coronal diameter × 3 mm in apical diameter × 4 mm in depth), with measurements verified using a digital caliper (Figure 1). The semi-indirect restoration technique was simulated by molding each standardized cavity with addition-cured silicone (Panasil Putty Soft, Ultradent Products Inc., South Jordan, UT, USA). The same molding protocol was used for all specimens, and the silicone material was inserted to completely reproduce the internal cavity geometry. After polymerization of the impression material, the molds were filled with die model silicone (VOCO GmbH, Cuxhaven, Germany), allowing the fabrication of a standardized working model for each cavity. The composite restorations were then fabricated on these models using Filtek Z350 XT composite resin (3M Oral Care, St. Paul, MN, USA), inserted by the incremental technique. Each increment was light-cured for 20 seconds, with the light-curing tip positioned perpendicular to and in direct contact with the restoration surface/model, standardizing the curing distance at 0 mm. After fabrication, each restoration was checked in its corresponding bovine tooth cavity to verify passive seating and marginal adaptation before cementation. Restorations presenting visible defects, bubbles, or lack of passive adaptation were excluded and remade.

The restorations were then cemented into the prepared cavities of the bovine incisors. Cementation was performed according to the clinical protocol recommended for semi-indirect restorations by each manufacturer. Light-curing was performed using a light source (VALO™ Cordless, South Jordan, USA) with an irradiance of 1000 mW/cm^2, according to the manufacturer's specifications. The irradiance was not verified with a radiometer during the experiment; therefore, this information was added as a methodological limitation. After seating the restoration, excess cement was carefully removed, and light curing was performed for 20 seconds. All specimens were polished using rubber cups and bristle brushes. After restoration and polishing, the specimens were stored in distilled water at 37°C for 24 hours before the push-out test.

Adhesive and cementation protocols

For the RelyX ARC group, the dentin surface was treated with Single Bond Universal adhesive

Table II - Study design

Group	Resin cement	Adhesive protocol
G1	Conventional RelyX ARC – 3M	Single Bond Universal adhesive – 3M
G2	Self-adhesive RelyX U200 – 3M	None
G3	Conventional NX3 – Kerr	Optibond S adhesive – Kerr
G4	Self-adhesive Maxcem Elite – Kerr	None

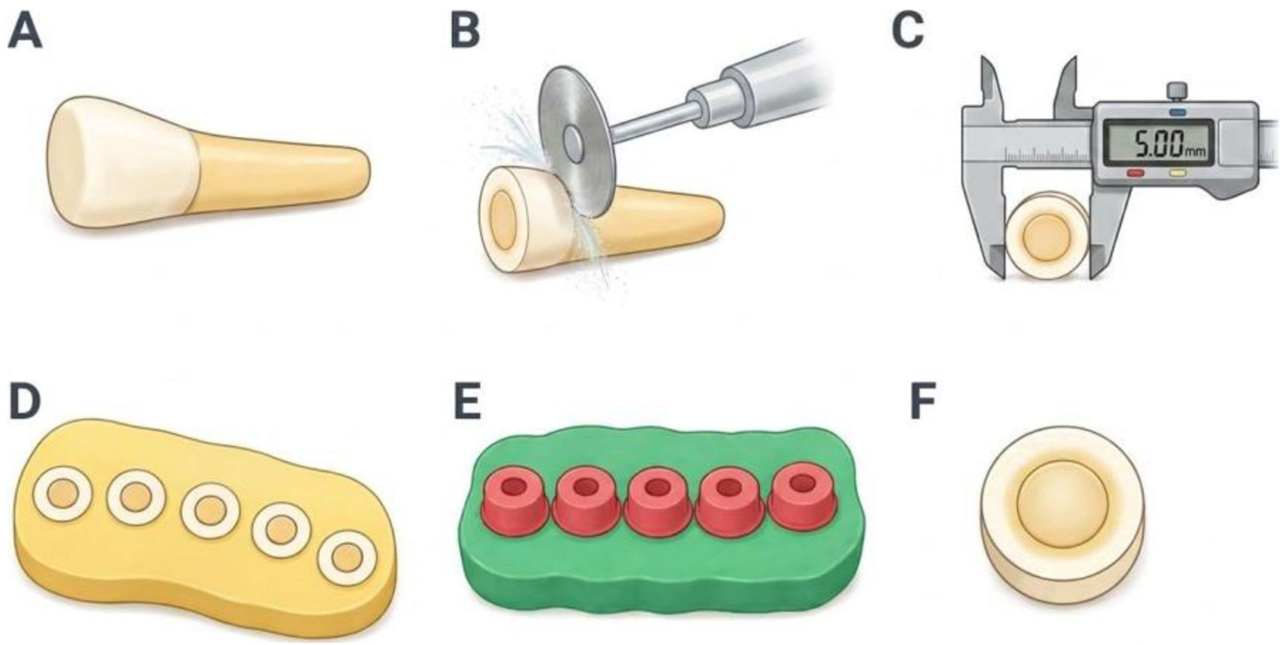

FIGURE 1 - Sample making process. **A.** Selection of bovine incisors without cracks or structural defects; **B.** Crown cutting with a diamond disc; **C.** Verification of specimen measurements with a digital caliper; **D.** Molding of specimens in groups of 5 with addition silicone (Panasil Putty Soft); **E.** Obtaining the model for fabrication of the semi-indirect restoration with silicone for die models; **F.** Cementation of the resin piece in the cavity of all specimens.

(3M Oral Care, St. Paul, MN, USA). The adhesive was actively applied to the dentin surface for 20 seconds, gently air-thinned for 5 seconds to evaporate the solvent, and light-cured for 10 seconds. The RelyX ARC cement was then manipulated according to the base/catalyst system and applied to the internal surface of the restoration. The restoration was seated into the cavity with standardized digital pressure, excess cement was removed, and light curing was performed for 20 seconds.

For the RelyX U200 group, no previous acid etching or adhesive application was performed, since this material is a self-adhesive resin cement. The cement was manipulated and applied directly to the internal surface of the restoration, which was then seated into the cavity with standardized digital pressure. Excess cement was removed, and light curing was performed for 20 seconds.

For the NX3 group, the dentin surface was treated with OptiBond S adhesive (Kerr, Orange, CA, USA). The adhesive was applied to the dentin surface using a disposable applicator with a brushing motion, gently air-thinned, and light-cured before cement placement. The NX3 resin cement was then applied to the internal surface of the restoration, which was seated into the prepared cavity with standardized digital pressure. Excess cement was removed, and light curing was performed for 20 seconds.

For the Maxcem Elite group, no previous acid etching or adhesive application was performed, since this material is a self-adhesive resin cement. The cement was dispensed directly onto the internal surface of the restoration, the restoration was seated into the cavity with standardized digital pressure, excess cement was removed, and light curing was performed for 20 seconds.

For all groups, the restorations were maintained in position during the initial cementation period to avoid displacement, and the same light-curing protocol was used to standardize polymerization among specimens.

Push-out test

Data were obtained through laboratory evaluation using the push-out mechanical test. To measure bond strength, a universal testing machine (Instron 4411, Instron, England, United Kingdom) equipped with a support device containing a central orifice was used. The specimens were positioned with the smaller base facing upward. A metallic plunger with a spherical tip was attached to the machine and applied to the restoration at a constant speed of 0.5 mm/min until complete displacement of the specimen occurred. The force required to promote the dislodgment of the restoration was recorded at the moment of fracture, and the results were expressed in kilogram-force (Kgf) and Newtons (N).

Statistical analysis

A descriptive analysis was performed, and the results were presented as mean values and standard deviations. The Shapiro-Wilk test ($p \geq 0.095$) and Levene's test ($p = 0.166$) were applied to verify the assumptions of normality of the data and homogeneity of variances, respectively. Inferential analysis was conducted using one-way Analysis of Variance (ANOVA), followed by Tukey's post hoc test, with a significance level of 5%. All analyses were performed using SPSS (Statistical Package for the Social Sciences) version 22.0 for Windows.

RESULTS

A significant effect of cement type on bond strength was observed ($p = 0.015$). The self-adhesive cement RelyX U200 showed the highest bond strength values in MPa (Table III), differing significantly from Maxcem Elite, which exhibited the lowest values. The conventional cements RelyX ARC and NX3 presented intermediate values, with no statistically significant differences between them.

DISCUSSION

The null hypothesis was partially rejected, since a significant difference in bond strength was observed between one of the self-adhesive resin cements and the other groups evaluated. The study employed a specific protocol for the semi-indirect technique, simulating the entire clinical molding procedure that occurs in the real clinical environment, thus contributing to the literature by providing evidence that more accurately reflects dental practice [17].

In existing studies on resin cements in the literature, it is possible to observe more in vitro results on intraradicular retainers than on semi-indirect or indirect restorations. This methodological gap motivated the present study, which aimed to innovate by mimicking the intraoral molding procedure [18-23].

The conventional resin cement, which uses an intermediate bonding agent, allows for the involvement of dentinal collagen fibrils, enabling better penetration of the cement into the demineralized dentin substrate [4]. The bonding depends on the hybrid layer, the restoration, surface roughness, and interface integrity; thus, the presence of the adhesive enables a bridge between the hydrophilic tooth and the hydrophobic cement, improving the outcomes [6]. However, factors involved in restorative rehabilitation through conventional cementation (restorative material, operator experience, and adhesive system quality) may limit its clinical success [18].

Table III - Bond strength values (MPa)

Group	Bond strength (MPa)
Self-adhesive cement RelyX U200 – 3M	4.01 ± 1.28^{a}
Conventional cement RelyX ARC – 3M	3.70 ± 0.70^{ab}
Self-adhesive cement Maxcem Elite – Kerr	2.73 ± 0.95^{b}
Conventional Cement NX3 – Kerr	3.06 ± 1.08^{ab}

Note: Different letters indicate statistically significant differences among groups according to Tukey's test ($p \leq 0.05$). MPa values were calculated by dividing the maximum load in N by the bonded area in mm^2.

The self-adhesive resin cement features a less technique-sensitive procedure by eliminating additional pre-treatment steps to the substrate [24]. The chemical composition of the self-adhesive cement and its inorganic polymeric characteristics can result in different mechanical properties, such as elastic modulus, compressive strength, and tensile strength [25]. The simplification of surface treatment for self-adhesion is the result of the last ten years of research, which has enabled clinicians to reduce dependence on operator skills as well as on specific equipment [7]. Similar findings were reported by Pacheco [12], who demonstrated that compositional modifications in a self-adhesive resin cement, such as the incorporation of TiO_2 nanotubes, significantly increased bond strength and radiopacity, reinforcing that the performance of these materials depends directly on their formulation and polymerization mode.

This study showed different outcomes for the groups of resin cements used. Despite similar values among some groups, the self-adhesive resin cement RelyX U200 (3M) demonstrated the highest bond strength values in MPa (Table III). The superior performance of RelyX U200 may be attributed to its self-adhesive mechanism, which relies on an optimized balance between acidic monomers and filler content. This promotes effective initial demineralization followed by rapid neutralization, enhancing polymerization and mechanical stability. Although the specific acidic monomers in RelyX U200 are not disclosed by the manufacturer, they are known to be methacrylate-based and capable of inducing superficial demineralization. These monomers are subsequently neutralized through ionic interactions with calcium from hydroxyapatite and alkaline components of the cement, leading to pH stabilization and contributing to improved interfacial bonding [26]. Other studies that employed the push-out test obtained similar results, as seen in the data from Santi [27], who compared different resin cements and reported good stability results for self-adhesive cements. Additionally, the self-adhesive cement reached a neutral pH rapidly, within 48 hours after cementation, which enables neutralization reactions and improves mechanical properties in the cementation of glass fiber posts [28].

The results of the self-adhesive cement from 3M (RelyX U200) showed better bond strength, although with a higher standard deviation compared to the self-adhesive cement Maxcem Elite (Kerr), a fact possibly related to early fracture of the specimens in one of the groups, or potentially associated with the material's setting curve [29]. Each cement has a specific working time that allows for manipulation and cementation, which may be a factor in the variation of the cement–dentin bond and may represent a limitation depending on the operator.

In the study by Calheiros-Lobo [30], the use of self-adhesive cements in semi-indirect restorations was observed, showing good mechanical characteristics and simplified cementation procedures due to the elimination of acid etching and/or adhesive system steps. Additionally, factors related to a slight increase in acidic pH promoted self-adhesion and improved resistance to hydrolysis-induced degradation. This effect is attributed to the presence of acidic monomers (multifunctional acidic methacrylates), which demineralize the dental substrate and enable infiltration of the luting agent into the conditioned area, resulting in micromechanical retention [31].

The two conventional cements, Rely ARC (3M) and NX3 (Kerr), performed better compared to the self-adhesive cement Maxcem Elite (Kerr), suggesting the superiority of conventional cements over self-adhesive cements in comparisons between different manufacturers [4,32,33]. Adhesion to dentin represents a determining factor for long-term clinical success, due to the integrity of the adhesive interface. Laboratory studies affirm that conventional resin cements promote greater and more stable adhesion to dentin, since the formation of the hybrid layer occurs by modifying the smear layer and demineralizing the underlying dentin, exposing the three-dimensional collagen layer that will be infiltrated by cement monomers [4,34,35].

The literature suggests that adding dental acid etching to the self-adhesive cementation protocol can improve strength results [24,36,37]. The enamel bonding mechanism is established by the micromechanical interlocking provided by resin penetration into the acid-etched enamel. Phosphoric acid etching prior to the adhesive system improves enamel bond strength [38,39]. Therefore, the use of self-adhesive cement combined with selective enamel etching can be recommended, resulting in more efficient adhesion.

The literature also indicates that clinical performance is strongly affected by how dentin is managed before cementation and by the type of luting agent used. Yousif and Kadhim [40] reported that immediate dentin sealing significantly reduced marginal gaps in lithium disilicate overlay restorations, and that dual-cure resin cement and flowable composite produced better marginal adaptation than preheated composite, while all groups remained within clinically acceptable limits. It can be stated that the results of this in vitro test are similar to those reported in the clinical study by Canatan [41], who found no statistical difference in survival of cemented CEREC Omnicam nanoceramic resin inlay/onlay restorations. Their results demonstrate clinical success for anatomical form, marginal integrity, and patient satisfaction with both types of luting agents. These clinical observations are similar to the results of Taschner [42], who evaluated self-adhesive cement in glass-ceramic inlays with a 2-year follow-up.

An analysis of clinical tests shows that despite there being no significant differences between conventional and self-adhesive resin cement, multi-stage cementation is technically sensitive and prone to errors, and may lead to total detachment of the restorative piece [43]. However, there is a lack of comparative clinical data from observation periods of more than one year between semi-indirect cementing agents.

The study was conducted with entirely manual sample preparation and two operators, which can be considered a risk factor for bias. The main limitation is the use of bovine teeth, which simulate human teeth to some extent. Furthermore, saliva immersion and analysis of bond strength at different times would provide more data for better comparison of restoration success. Aging protocols could simulate clinical degradation and allow prediction of material performance. In this study, bond strength was assessed using a push-out test. However, other methodologies, such as microtensile bond strength testing, may also be employed. A limitation of this study is that no formal statistical power analysis was performed to determine the sample size. Although the number of specimens per group was based on previous studies with similar methodologies, the absence of a priori calculation may affect the robustness and generalizability of the results. Another limitation of the study was that the irradiance of the light-curing unit was not verified with a radiometer during the experimental procedures.

CONCLUSION

Differences in cement formulation impacted bond strength outcomes. Although RelyX U200 yielded the highest values, Maxcem Elite presented reduced bond strength compared with conventional cements, suggesting that performance is not uniform within self-adhesive systems.

Data availability

The data supporting the findings of this study are available upon request to the corresponding author.

Author's Contributions

SAMC, WKMS, LMRM: Conceptualization. SAMC, LMRM: Methodology. MLS: Software. TOC, JVCL, RCCG: Validation. CCA, GCB, LMRM: Formal Analysis. SAMC, WKMS: Investigation. LMRM: Resources. MLS, TOC, RCCG: Data Curation. SAMC, WKMS: Writing-Original Draft Preparation. SAMC, JVCL, RCCG, LMRM: Writing-Review & Editing. CCA, GCB: Visualization. RCCG, LMRM: Supervision. SAMC, LMRM: Project Administration. LMRM: Fundraising.

Conflict of Interest

No conflicts of interest declared concerning the publication of this article.

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Regulatory Statement

Not applicable.

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