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Bond strength of bulk-fill resin composites: the effect of cavity preparation and aging

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Aim: Evaluating the resin-dentin bond strength of Class II conventional and bulk-fill composite restorations, using different cavity sizes before and after aging. **Methods:** Seventy-five human molars were distributed into groups according to the buccolingual width of the cavities, conservative (n=25) and extended (n=50). They were divided according to the restorative material: conventional (Z100/control group) or bulk-fill resin composites (Filtek Bulk Fill/FBF; Tetric N Ceram Bulk Fill/TNCBF; Filtek Bulk Fill Flow/FBFF; Surefill SDR flow/SDR). The restored teeth were sectioned on sticks (n=50 per restorative materials + width cavities group), half were stored in Water/Ethanol 75% for 30 days and the other half were submitted to the immediate microtensile bond strength (μTBS) test. Data were analyzed applying the Three-Way Analysis of Variance (ANOVA), Bonferroni test, test t, and Weibull analyses ($p<0.05$). **Results:** SDR and FBF presented lower μTBS values for extended preparation when compared to the conservative preparation, before aging. After aging, only for the FBFF, a decrease in the μTBS values was observed. Comparing the μTBS values, before and after aging, the SDR demonstrated lower μTBS values after aging when the conservative cavity was used. A decrease in the μTBS values was observed for the Z100, the FBF and, the FBFF, after aging, when the extended cavity was used. **Conclusion:** The effect of cavity preparation and aging on the resin-dentin of Class II is material dependent. Most of the bulk-fill resin composites evaluated presented a similar performance to the conventional resin composites for all the conditions of this study.

Keywords: Composite resins. Tensile strength. Aging. Dental cavity preparation.

Introduction

Bulk-fill resin composites have been used by clinicians to simplify dental operative procedures. These resin composites were introduced to be inserted in a single increment of 4–5mm, being an attractive alternative for posterior restorations[1]. Manufactures have applied different strategies to formulate a material presenting better light transmission and reduced polymerization stress. To improve the depth of polymerization, alternative and more reactive photoinitiators, as well as lower filler concentrations, are used[2-5]. Modified monomers, such as novel stress-relieving monomers and methacrylate monomers, containing a third reactive site, have been incorporated into the bulk-fill resin composites to reduction of the polymerization stress[2-5]. Two types of bulk-fill composites viscosity are available: low-viscosity and high-viscosity. Low-viscosity bulk-fill resin composite is indicated to replace dentin, filling most of the cavity, followed by capping with the conventional resin composites. Using high-viscosity bulk-fill resin composites, only one increment can be applied and sculpt the occlusal surface simultaneously[6].

Some factors, including polymerization shrinkage of the resin composites, may negatively affected clinical durability of resin composite restorations[7]. Polymerization shrinkage stress may create tensile stress on the adhesive tooth restoration interface, affecting the bond strength and the marginal integrity of restorations[8]. As a result, some clinical consequences such as post-operative hypersensitivity, marginal discoloration, cohesive tooth fractures at the margins, recurrent caries and pulpal inflammation can be observed[9]. To provide better sealing for the cavity margins, bulk-fill resin composites have been developed. These resin composites seem to be an interesting option to enhance the resin-adhesive bonding to the tooth structure in regions without adequate marginal integrity, such as the cervical margins of class II cavities[10]. Micro-leakage and bond strength tests, associated with artificial aging, have been used to investigate marginal integrity and bonding quality to tooth of resin composite-restorations[11]. Also is suggested that artificial aging has influence on the integrity tooth-composite interface[12].

Controversial results about the bulk-fill resin composites presenting better sealing of the cavity margins and adequate bond strength to the dental substrate have been reported in the literature[5,12,13]. Consequently, clinicians are still insecure about the use of this new class of materials in the clinical practice[11]. Therefore, this *in vitro* study aimed to evaluate the effect of the cavity size and artificial aging on the resin-dentin bond strength of Class II conventional and bulk-fill composite restorations. The following experimental hypotheses were tested: 1) conservative cavity size will have better resin-dentin bond strength of Class II conventional and bulk-fill composite restorations than the extended cavity; 2) artificial aging will have effect on the resin-dentin bond strength of Class II conventional and bulk-fill composite restorations; 3) The resin-dentin bond strength of the conventional and the bulk-fill composite restorations will be comparable.

Materials and Methods

Tooth Selection and Experimental Groups

Seventy-five healthy human third molars were used in this study after the approval from the Research Ethics Committee of the University of Paraiba (protocol n. 2.048.942). The teeth inspection was performed using an optical microscopy to select only teeth free from caries and with no cracks or developmental defects. After the selection, the teeth were cleaned, stored in a 0.2% thymol solution and used within one month after extraction. All tooth roots were embedded in self-curing acrylic resin. Initially, the teeth were randomly distributed into groups according to the combination of the buccolingual width, conservative (n=50) and extended (n=25). This difference in the number of teeth between the groups is because conservative preparations provide smaller number of toothpicks than extended group. This step is better described below. A second distribution was made according to the resin composite used. Three types of bulk-fill resin composites were used: Filtek Bulk Fill, 3M ESPE dental products (FBF), Tetric N Ceram Bulk Fill, Ivoclar Vivadent, (TNCBF), Filtek Bulk Fill Flow 3M ESPE dental products (FBFF), Surefil SDR Flow, DENTSPLY (SDR) and a conventional resin composite Z100, 3M ESPE dental products, (Z100). Tested materials are in the table 1.

Table 1. Tested Materials

COMPOSITE	COMPOSITION	BATCH NUMBER
Z100 (3M)	Bis-GMA, TEGDMA, zirconia/silica with 71% weight by volume). Particle size: 0.01 to 3.5 μm (average: 0.6 μm).	1822500253
FILTEK BULK FILL (3M)	AUDMA, UDMA and 1,12-dodecane-DMA. Zirconia (4-11 nm) and silica (20 nm) that can be aggregated and agglomerated or not. Iterbium trifluoride from agglomerated particles (100 nm). 76,5% by weight (58.4% by volume).	N920657
TETRIC N CERAM BULK FILL (IVOCLAR)	Bis-GMA, bis-EMA and UDMA (19-21% by weight) and 75-77% by weight (53-55% by volume) inorganic particles (average: 0.6 μm). The filler consists of barium glass, prepolymer, ytterbium trifluoride and mixed oxides. The particle size of the inorganic fillers is between 0.04 and 3 μm.	w94624
FILTEK BULK FILL FLOW (3M)	BIS-GMA, UDMA, BIS-EMA 6 and procrylat. Ytterbium trifluoride and zirconia/silica with 64.5% by weight (42.5% by volume). Particle size, respectively: 0.1 to 5.0 microns and 0.01 to 3.5 μm.	1531700424
SURIFIL SDR (DENTSPLY)	EBPADMA, TEGDMA, camphoroquinone (cq) as Photoinitiator; photoaccelerator; hydroxy toluene Butylate (bht); uv stabilizer; titanium dioxide; Fluorescent agents. Particle size: 20nm to 10μm, and the charge content by volume is about 47.3%.	150827

Specimen Preparation and Restorative Procedure

The cavities were prepared according to standardized dimensions: occlusal box deep was 3mm and mesiodistal length at the bottom of the proximal box was 5mm. The proximal box (mesially and distally) was 5mm deep with margins located 1mm below the cemento-enamel junction. Each cavity had the inner walls perpendicular to the top and bottom surfaces, with round angles defined by the bur's shape. Teeth were distributed into two groups according to the buccolingual width: conservative cavity (2mm wide in the buccolingual direction) and extended cavity (4mm wide in the buccolingual direction). The cavities were prepared using a diamond bur under water cooling (#1150, KG Soresen; Barueri, SP, Brazil). The two-step etch and rise adhesive Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA) was applied following the manufacturers' instructions. After the adhesive application, a metal matrix band was placed, and the teeth were restored according to the restorative material: conventional or bulk-fill composite resin. The conventional composite (Z100-3M ESPE St. Paul, MN, USA) was placed in a 1–1.5mm thick horizontal layer, applying an incremental technique. Each increment was separately light cured for 20 s (800 mW/cm^2, Emitter C, SCHUSTER, Santa Maria, RS, Brazil). The bulk-fill resin composites were applied in a 3.5 to 4-mm layer and then, light cured, following the manufactures instructions. The restored teeth were stored at 37 °C (±1°C) in distilled water/ethanol 75% for 24 hours. A single operator performed all procedures.

After storage time, the proximal box of restorations was longitudinally sectioned in the mesiodistal and buccolingual directions across the bonded interface. The sections were executed using a slow-speed with a diamond saw in a Lab-cut 1010 machine (Extec, Enfield, CT, USA) underwater cooling to obtain resin-dentin sticks with a rectangular cross-sectional area of approximately 1mm^2. For each group (conservative and extensive of each restorative material)., fifty sticks were obtained from proximal boxes. Twenty-five sticks were submitted to microtensile bond strength testing and the other half was stored at 37 °C (±1°C) in distilled water/ethanol 75% for 30 days.

Microtensile bond Strength Testing (µTBS)

The µTBS testing was performed with a crosshead speed of 5 mm/min using a universal testing machine (Odeme, Luzerna, SC, Brazil). The sticks were attached to a modified microtensile testing device with cyanoacrylate resin (Super Bonder, Loctite; São Paulo, SP, Brazil). To obtain µTBS (MPa) values, the measured force (N) was divided by the individual bonded area (mm^2). When sticks failed while being sectioned or attached to the tester, they were excluded from the study.

The failure mode was evaluated at 200x using light stereo microscopy (HMV-2, Shimadzu, Kyoto, Japan). The failure modes were categorized as follow: cohesive failure in the adhesive (type I), cohesive failure in the dentin (type II), cohesive failure in the hybrid layer (type III), mixed failure (cohesive failure in the adhesive and in the hybrid layer- type IV), cohesive failure in the resin composite (type V).

Statistical Analysis

The µTBS data were subjected to the Kolmogorov-Smirnov test to verify the normality. Then, the data were analyzed using a three-way Analysis of Variance (ANOVA) and post hoc Bonferroni test, as well as the test t at 0.05 level of significance. To evaluate the reliability of the bond strength, the Weibull analysis was applied for each group. The Weibull moduli (shape parameter) (slope of the line relating applied stress and the probability of specimen failure, m) were calculated, applying maximum likelihood estimation. The 95% upper and lower confidence intervals were calculated using the likelihood ratio (MINITAB 17.0, State College, Pennsylvania, USA).

Results

Comparing the μTBS values of the conservative and the extended cavities, the SDR bulk-fill composite (p=0,03) and the Filtek Bulk Fill flow (p=0,04) presented lower μTBS values for the extended preparation before artificial aging. On the other hand, a decrease in the μTBS values was observed only for the Filtek Bulk Fill flow (p=0,01) after aging (Table 2).

Table 2. Means and standard deviation of μTBS values for resin composites studied (Mpa)

STORAGE	COMPOSITE	CONSERVATIVE	EXTENDED
BEFORE	Z100	31,48 (13,29) Aa	29,22 (9,61) Aa
	FBF	30,13 (14,60) Aa	32,36 (13,92) Aa
	TNCBF	29,48 (14,63) Aa	28,90 (10,44) Aa
	FBFF	33,67 (16,11) Aa	25,06 (11,31) Ab
	SDR	35,36 (16,24) Aa	27,24 (10,89) Ab
AFTER	Z100	28,28 (12,34) Aa	21,69 (10,83) Bb
	FBF	23,04 (9,73) Ba	24,68 (9,98) Ba
	TNCBF	24,39 (9,77) Aa	26,19 (12,90) Aa
	FBFF	27,09 (13,77) Aa	18,84 (8,73) Bb
	SDR	24,77 (13,56) Ba	22,52 (9,77) Aa

Different letters represent significant differences ($p < 0.05$): uppercase within columns (for before and after storage independently); lowercase within rows.

Table 2 shows the results of the μTBS values, comparing the values before and after artificial aging for conservative and extended cavities. Regarding the conservative cavity, the SDR bulk-fill (p=0,01) composite demonstrated lower μTBS values after 30 days- storage in distilled water/ethanol. A decrease in the μTBS values was observed for the Z100 (p=0,01), the Filtek Bulk Fill (p=0,03), and the Filtek Bulk Fill flow (p=0,03) after artificial aging when the extended cavity was used. No significant difference between the resin composites in the μTBS values was noted, before or after the aging process. Table 3 shows the results of the failure mode analysis after bond testing, revealing that most of the failures were mixed fractures for all experimental conditions.

The results of the Weibull analysis are showed in Table 4 and Figure 1. No difference in the *m* values was observed for all experimental groups and conditions.

Table 3. Classification of failure modes (%) before and after storage

BEFORE STORAGE										
	EXTENDED					CONSERVATIVE				
	FAILURE MODES									
	I	II	III	IV	V	I	II	III	IV	V
Z100	0%	0%	0%	84%	16%	0%	0%	0%	84%	16%
FBF	16%	0%	0%	48%	36%	16%	0%	0%	48%	36%
TNCBF	4%	4%	0%	80%	12%	4%	4%	0%	80%	12%
FBFF	12%	0%	0%	80%	8%	12%	0%	0%	80%	8%
SRD	4%	12%	0%	76%	8%	4%	12%	0%	76%	8%
AFTER STORAGE										
	EXTENDED					CONSERVATIVE				
	FAILURE MODES									
	I	II	III	IV	V	I	II	III	IV	V
Z100	8%	0%	0%	67%	16%	0%	0%	0%	80%	20%
FBF	16%	0%	0%	48%	36%	4%	0%	0%	76%	20%
TNCBF	12%	0%	0%	76%	12%	8%	4%	0%	72%	16%
FBFF	4%	0%	0%	80%	16%	8%	0%	0%	82%	10%
SRD	9%	0%	0%	79%	12%	7%	0%	0%	83%	12%

Type I - cohesive failure in adhesive; Type II - cohesive failure in detin; Type III - cohesive failure in hybrid layer; Type IV - mixed failure (cohesive failure in adhesive and hybrid layer);Type V - cohesive failure in resin composite.

Table 4. Weibull moduli (m) values, among the experimental groups comparing the resin composites for conservative and extended cavity before (24 h) and after storage (30 days).

STORAGE TIME	COMPOSITE	CONSERVATIVE	EXTENTED
24 HOURS	Z100	2.63 (1.93-3.58)^{Aa}	2.85 (2.30-3.54)^{Aa}
	FBF	2.26 (1.65-3.08)^{Aa}	2.37 (1.90-2.96)^{Aa}
	TNCBF	2.18 (1.60-2.96)^{Aa}	2.52 (2.04-3.13)^{Aa}
	FBFF	2.27 (1.73-3.31)^{Aa}	2.16 (1.75-2.66)^{Aa}
	SDR	2.39 (1.68-3.06)^{Aa}	2.35 (1.88-2.93)^{Aa}
30 DAYS	Z100	2.68 (1.98-3.64)^{Aa}	2.36 (1.90-2.94)^{Aa}
	FBF	2.63 (1.92-3.61)^{Aa}	2.76 (2.21-3.44)^{Aa}
	TNCBF	2.73 (2.02-3.69)^{Aa}	2.35 (1.90-2.90)^{Aa}
	FBFF	2.13 (1.56-1.91)^{Aa}	2.35 (1.90-2.90)^{Aa}
	SDR	1.99 (1.49-2.66)^{Aa}	2.28 (1.92-2.72)^{Aa}

Means followed by same uppercase letters in the same row and column indicate no statistically significant differences between the groups (*p* >0.05).

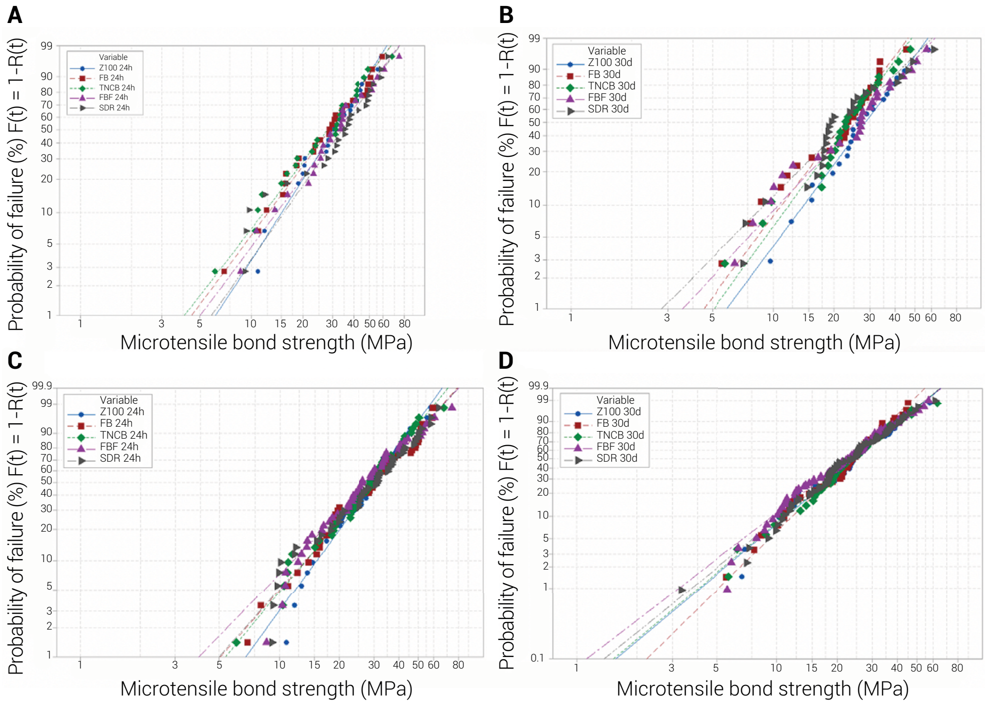

Figure 1. Weibull distribution plots of microtensile bond strength data for the experimental groups comparing the resin composites. FBF-Filtek bulk-fill; TNCBF- Tetric N ceram bulk-fill; FBFF-Filtek bulk-fill flow; SDR- SDR flow. (A) Conservative cavity group after 24 hours storage; (B) Conservative group after 30 days storage; (C) Extended cavity group after 24 hours storage; (D) Extended cavity group after 30 days storage.

Discussion

Bulk-fill composites have been developed to be inserted in increments of up to 4mm in thickness without compromising the mechanical properties and marginal quality of the restoration[14]. The performance of these resin composites in terms of bond strength to dentin is still unclear, mainly when those composites are used to restore large cavities. In this study, the resin-dentin bond strength of Class II high viscosity and flowable bulk-fill resin composites restorations was evaluated, using different cavity sizes. Previous research studies verified that large cavities were not favorable for bonding composites to tooth material, being an incremental technique more effective in those cavities[15]. According to the current study, the SDR and the Filtek Bulk Fill resin composites restorations demonstrated lower μSBS values in extended cavity preparation when compared to the conservative cavity before artificial aging. After artificial aging, a decrease in the μSBS values was observed only for the Filtek Bulk Fill flow. Thus, the first experimental hypothesis was rejected.

Polymerization shrinkage stresses developed in the adhesive interface of restorations can affect resin-dentin bond strength when composites' contraction is restricted by the cavity walls[16,17]. Several factors, including material composition, composite resin placement technique, geometry, and cavity extension can influence the magnitude of the polymerization stress[18-20]. This study showed the negative influence of extended cavity size on the bond strength of some bulk-fill resin composites to the dentin (table 2). These results are not following previous study[8]. This fact can be related to the difference in cavity configuration and testing methodology. Regarding the influence of artificial aging in the bond strength to the dentin, results demonstrated a significant influence of artificial aging (distilled water/ethanol) on the resin-dentin strength of Class II bulk-fill composite restorations. Hence, the results of this study lead to the rejection of the second experimental hypothesis. A decrease in the μSBS values for the SDR bulk-fill composite (conservative cavity), the Z100, the Filtek Bulk Fill and the Filtek Bulk Fill flow (extended cavity) was observed after artificial aging. This may be attributed to hydrolytic action of distilled water/ethanol on resin composite and the adhesive interface between the adhesive system and the resin composite, yielding a degradation of polymeric matrix[21].

Modifications in the matrix and filler of bulk-fill resin composites were made to increase their translucency and decrease the shrinkage stress. An increase in the filler size and the addition of more reactive photoinitiators are strategies used to allow greater light transmission with depth)[2-5]. Regarding shrinkage stress, the inclusion of proprietary stress reliever molecules and polymerization modulators seems to decrease the shrinkage stresses generated during resin polymerization[22]. Probably, the strategies used by bulk-fill manufactures explain the results of this study, in which conventional and bulk-fill composite restorations showed similar μSBS values in all studied conditions, agreeing with other studies[8]. Therefore, the third was rejected. Additional studies showed that bulk-fill resin composites presented better results of bond strength to the dentin than conventional composites for class II[23,24]. Systematics reviews of laboratory studies have shown similar or better performance of bulk-fill materials compared to the traditional composite resins in terms of polymerization stress, cusp deflection, marginal gap, degree of conversion, flexural strength, and fracture strength[25,26]. Furthermore, systematic review and meta-analysis of clinical trials have revealed no differences in the performance of bulk-fill and conventional materials after 01 to 10 years of follow up[27,28]. Thus, it seems that bulk-fill resin composites seem to be a suitable alternative to conventional layered resin composites when used in a 4 -5mm single-increment (bulk-fill technique)[29].

The bond strength values were analyzed using the Weibull statistic[30]. The bonding effectiveness to dentin and ceramics can be assessed by Weibull survival analysis[31]. Probably, high values of modulus mean that the bonding procedure is more reliable[32]. The Weibull analysis revealed that similar *m* values were obtained for all groups. This finding suggests that the bond strength between bulk-fill resin composite to dentin present equal reliability than conventional resin composites. Considering the analysis of fracture mode, mixed failure was the predominant fracture pattern for all experimental groups. These results agree with other studies[8,33], suggesting that the hybrid layer was formed, but was fractured due to concentrated tension at the adhesive interface[34-37].

The results of this research study indicate that the type of cavity size (conservative or extended) and artificial aging negatively influenced the bond strength of some bulk-fill resin composites to the dentin. Moreover, the studied bulk-fill resin composites presented similar bonding effectiveness to the dentin than conventional resins for all experimental conditions. However, this *in vitro* study does not test all bulk-fill resin composites available in the market and does not reproduce intraoral conditions.

Therefore, further investigations, using different materials and conditions to simulate the buccal environment, are necessary to validate these findings.

Within the limitations of the current study, the following was concluded:

1. The type of cavity preparation affected the bond strength of the Filtek Bulk Fill Flow and the SRD Flow restorations before artificial aging and Filtek Bulk Fill flow restorations after artificial aging.

2. The bond strength of the Z100, the Filtek Bulk Fill and the Filtek Bulk Fill Flow restorations was influenced by artificial aging when an extensive preparation cavity was used. While the SRD Flow restorations bond strength was affected by the conservative preparation cavity.

3. The bond strength of most bulk-fill resin composite restorations was similar to conventional composite restorations regardless of the type of cavity preparation and artificial aging employed.

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Data availability

Datasets related to this article will be available upon request to the corresponding author.

Author Contribution

Caroline de Farias Charamba: Substantial contributions to the conception and design of the work, acquisition, analysis and interpretation of data for the work, drafting the work and revising it critically for important intellectual content, final approval of the version to be published, agreement to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

Larissa Dias Boson: contributed with the design of the article, acquisition and interpretation of data, drafting the article, final approval of the version to be published, agreement to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

Renally Bezerra Wanderley Lima: contributed in the acquisition of data, analysis

and interpretation of data, drafting the article and revising critically for important intellectual content, final approval of the version to be published, agreement to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

Rosangela Marques Duarte: contributed to conception and design of the article, acquisition of data and drafting the paper, final approval of the version to be published, agreement to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

Ana Karina Maciel de Andrade: contributed to conception and design, acquisition of data, and analysis and interpretation of data. Additionally, this author was important in drafting the article and revising it critically for important intellectual content. Final approval of the version to be published, agreement to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

All authors actively participated in the discussion of the manuscript's findings, and have revised and approved the final version of the manuscript.

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