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Original Article

Bottom/top hardness ratio and dentin bonding stability of conventional and bulk-fill resin composites

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Aim: To evaluate the bottom/top hardness ratio (B/T) and the dentin bonding stability of conventional and bulk-fill resin composites in high c-factor preparations. **Methods:** Regular conventional (Tetric N-Ceram – TNC, and Polofil Supra – PFS), regular bulk-fill (Tetric N-Ceram Bulk fill – TBF, and Admira Fusion X-tra – AFX), and low viscosity bulk-fill resin composites (Tetric N-flow – TNF, and X-tra Base – XTB) were used to restore 180 dentin conical preparations. The specimens were randomly distributed in 12 groups (n = 15) according to the resin composites and storage time-points (24 h and six months) tested. After 24 h storage, all specimens were subjected to the bottom/top hardness ratio analysis. Then, the push-out bond strength test was performed in half of the specimens and the other half were maintained for six months on water storage before testing. The failure modes were analyzed in a stereomicroscopic. The data were analyzed statistically using one- and two-way ANOVA and Tukey post-test ($p < 0.05$). **Results:** There were no statistically significant differences for the bottom/top hardness ratio among the resin composites ($p > 0.05$). Regardless of the storage time-point, regular bulk-fill resin composites showed the highest bond strength values statistically ($p < 0.05$). Only conventional resin composites showed statistically lower bond strength values at six-month storage ($p < 0.05$). Adhesive failures were more predominant for low-viscosity bulk-fill resin composites. **Conclusion:** Although the DoC was not affected by different materials tested, only bulk-fill resin composites did not present dentin bond strength loss after six-month of water storage.

Keywords: Dental bonding. Longevity. Composites resins. Dental materials.

Introduction

The stability of the adhesive interface is one of the primary factors for the success of restorations. The resin composite bonding to dental tissues must be stable to promote durability to the restoration. Bonding to dentin is a challenge due to its tubular conformation, water content, and organic components[1]. Thus, an effort has been made to find an adhesive protocol to promote greater dentin bonding stability to resin composites over time[2].

Mechanical properties such as the depth of cure are related to resin composites' dentin bonding performance[3-5]. An insufficient monomer conversion in the bottom of resin composite restorations can compromise their strength and durability due to the material's hydrolytic degradation[6,7].Thus, regardless of the resin composite type used, a well-polymerized material is required, which can be accessed using the bottom/top hardness ratio[3-5].

Low and regular viscosity bulk-fill resin composites were introduced in the market to become easier filling of high C-factor posterior tooth preparations with increments of up 4-5 mm[8,-9]. Low viscosity bulk-fill resin composites polymerized in 4-mm increments had lower shrinkage stress, higher bond strength and lower hardness than conventional resins composites[4]. Regular bulk-fill composite resins obtained similar or better results for bottom/top hardness ratio, marginal adaptation and interfacial nanoleakage compared to conventional composite resins[3]. However, the evaluation of dentin bonding stability to compare the performance of low and regular viscosity bulk-fill resin composites and its relation with bottom/top hardness ratio need further investigation.

Thus, this study aimed to evaluate the bottom/top hardness ratio (B/T) and the dentin bonding stability of conventional and bulk-fill resin composites with different viscosities. The null hypothesis tested in this study is that there will be no statistically significant differences between the materials for both properties analyzed.

Methods and materials

Experimental design

This research was characterized as an experimental *in vitro* study, whose composites used are listed in Table 1.

Table 1. Materials used in this study.

Material	Manufacturer	Type/viscosity	Shade	Lot	Composition
Tetric N-Ceram	Ivoclar (Liechtenstein)	Conventional/ Regular	A2	W91364	Urethane Dimethacrylate (≥10 - >25%), Ytterbium Trifluoride (≥10 - >20%), Bis-EMA (2.5 – 10%), Bis-GMA (≥2.5-<10%)
Tetric N-ceram Bulk fill	Ivoclar (Liechtenstein)	Bulk-fill/Regular	IVA	W91962	Bis-GMA (3 - <10%), Urethane Dimethacrylate (3 - <10%), Ytterbium Trifluoride (3 - <10%), Bis-EMA (3 - <10%)

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Continuation

Tetric N-flow	Ivoclar (Liechtenstein)	Bulk-fill/Low	IVA	W41268	Urethane Dimethacrylate (≥10 - <25%), Ytterbium Trifluoride (≥10 - <20%), Bis-GMA (10 – 25%), Triethylene Glycol Dimethacrylate (≥2.5 - <10%)
Polofil Supra	Voco (Germany)	Conventional/ Regular	A2	1810034	Bis-GMA (10-25%), Urethanedimethacrylate (5 – 10%), Triethylene Glycol Dimethacrylate (2.5 – 5%)
Admira Fusion X-tra	Voco (Germany)	Bulk-fill/Regular	E1	1736584	Organically Modified Silicic Acid (10 – 25%)
X- tra Base	Voco (Germany)	Bulk-fill/Low	A2	1742724	Bis-EMA (10 – 25%), Aliphatic Dimethacrylate (10 – 25%)
Single Bond Universal	3M ESPE (USA)	-	-	1724700342	Ethyl Alcohol (25-35%*), Bis-GMA (10 – 20%*), Silane Treated Silica (10 – 20%*), HEMA (5 – 15%*), Copolymer of Acrylic and Itaconic Acids (5- 15%*), Glycerol 1,3 Dimethacrylate (5 – 15%*), UDMA (<5%*), water (<5%*), Diphenyliodonium Hexafluorophosphate (<0.5%)

* Trade secret
Source: Material Safety Data Sheet (MSDS).

Specimens' preparation

A schematic representation of specimens' preparation and methods performed in this study is shown in Figure 1.

The technique described by Sousa-Lima et al.[10] (2017) guided the methodological aspects of this research. One hundred eighty healthy bovine incisors with no enamel cracks or structural defects were selected and decontaminated in an aqueous solution of thymol (0.1%) at 4°C for one week. Then, they were distributed in 12 groups (n = 15), according to the six resin composites (Table 1) and the two storage time-points tested (24 hours and six months). The roots' teeth were sectioned using a Diamond Flexible Disc (KG, Cotia, São Paulo, SP, Brazil) at the highest point of the cementitious junction and discarded. Subsequently, a parallel cross-section was made 5 mm above the first cut (in the incisal direction), through which a 5-mm thick specimen was obtained with a central void referring to the pulp cavity. To obtain 4-mm thick flat dentin specimens, #400 and #600 grit sandpapers (Labopol-21, Struers, Copenhagen, Denmark) were used to ground the upper (incisal) and lower (cervical) specimens' surfaces.

The central space referring to the pulp cavity was used for the cavity preparation with a tungsten carbide burs (Komet Inc., Lemgo, Germany) coupled to a handpiece under air-water cooling (Kavo, Joinville, Santa Catarina, Brazil), which was connected to in a standardizer device. The bur penetrated the center of the sam-

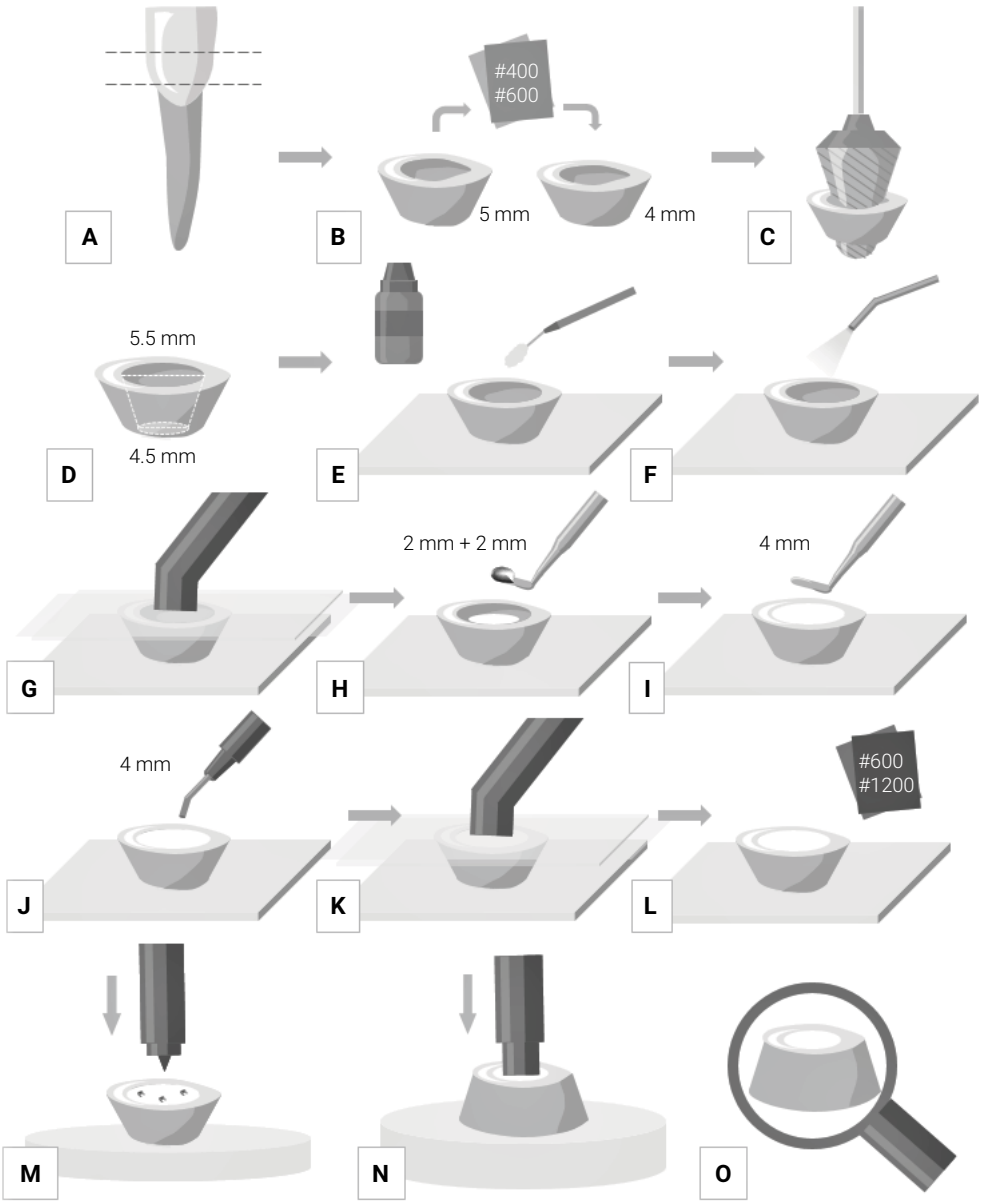

Figure 1. Bovine incisors were used. Sections at the highest point of the cementoenamel junction and 5 mm above were made (A). The specimens were ground with sandpapers to obtain 4 mm heigh (B). The cavity was prepared using a tungsten carbide bur (C), and a final preparation was obtained (D). The adhesive system was applied according to the manufacturer's recommendations (E-G), and the preparation was filled according to the resin composite used (H-K). The restorations were finished and polished (L) before submitting to the hardness (M) and bond strength (N) analyses. The failure modes were then analyzed (O).

ple, giving rise to an open, standardized conical cavity, with 5.5 mm upper diameter (incisal) x 4.5 mm lower diameter (cervical) and 4 mm thick. The bur was changed every 30 preparations.

After all the cavity preparations, excess water was blotted with absorbent paper, leaving the dentin surface visibly moist (wet bonding). The Single Bond Universal system (3M ESPE, St Paul, MN, USA) was applied according to the manufacturer's instructions, and its solvent was volatilized with an air spray for 5 s. The device tip was positioned on a glass slide to standardize the distance between the curing device and the upper specimen surface. The photoactivation was performed for 10 seconds with the Coltolux LED device (Coltène / Whaledent, Altstätten, Switzerland – 1200 mW/cm^2).

Each specimen was placed over a glass slide (1 mm thick) with the largest diameter opening upwards and the smallest diameter supported on the glass plate. The traditional resin composites were placed in two 2-mm thick increments separately photoactivated according to instructions of the manufacturer's with the Coltolux LED device (Coltène / Whaledent, Altstätten, Switzerland) during the time determined by the manufacturer (Table 2). In contrast, the low and regular viscosity bulk-fill composites were dispensed in single 4 mm increments and photoactivated according to the manufacturer's recommendations (Table 2). The curing device tip was placed over a glass slide (1 mm thick) on the resin composite surface to standardize the photoactivation distance for all resin composites. The restorations were finished with #600 and #1200 abrasive sandpapers coupled to a polishing machine (Labopol-21, Struers, Copenhagen, Denmark). Half of the samples were kept for 24 hours in distilled water at 37 ° C and the other half for six months.

Table 2. Operative protocol for each resin composite used in this study.

Resin composite	Number of increments	Increment thickness	Photoactivation* time per increment
TNC	2	2 mm	10s
TBF	1	4 mm	10s
TNF	1	4 mm	10s
PFS	2	2 mm	40s
AFX	1	4 mm	20s
XTB	1	4 mm	10s

*The device used in this research had a power > 1000 mW/cm² which was measured with a radiometer (Model 100, Kerr, Orange, CA, USA) for every eight specimens. TNF: Tetric N-Ceram; TBF: Tetric N-Ceram Bulk fill; TNF: Tetric N-flow; PFS: Polofil Supra; AFX: Admira Fusion X-tra; XTB: X-tra Base.

Bottom/top hardness ratio

The bottom-to-top hardness ratio was performed according to previous studies[3,5,11]. After 24 h water storage, the specimens were positioned on the base of a microhardness tester device (HMV-2T E, Shimadzu Corporation, Tokyo, Japan) and three Vickers indentations were performed in the central region of the top and bottom

surfaces of each specimen with a distance of 200 µm between them. A 50 g load was used for 30 s. The mean Vickers hardness number was obtained per surface, and the bottom/top hardness ratio was calculated.

Push-out bond strength test and failure modes

The bond strength was assessed after 24 h (n = 90) and six months (n = 90) of water storage through the push-out test in a universal testing machine (Microtensille OM150, Odeme, Joaçaba, Santa Catarina, Brazil). The specimens were placed on the device with its larger diameter (incisal) surface facing the metal base. A cylindrical 2.25 mm diameter metal tip pushed the smaller diameter (cervical) surface. It touched only the composite that filled the cavity, connected to the equipment's load cell (100 N) at a 0.5mm/min speed until the restoration rupture. The load required for the restoration failure was recorded in N and converted to MPa, according to the following equation:

$$MPa = \frac{N}{\pi(R + r)\sqrt{(h^2 + (R - r)^2}}$$

where 'R' is the radius of the larger base, 'r' is the radius of the smaller base, and 'h' is the thickness of the specimen.

After the test, the failure mode was examined using a dissecting microscope (Stereozoom; Bausch & Lomb, Rochester, NY, USA), using the following classification: adhesive between adhesive and dentin, cohesive in resin composite/dentin, and mixed (adhesive/cohesive) represented in Figure 2.

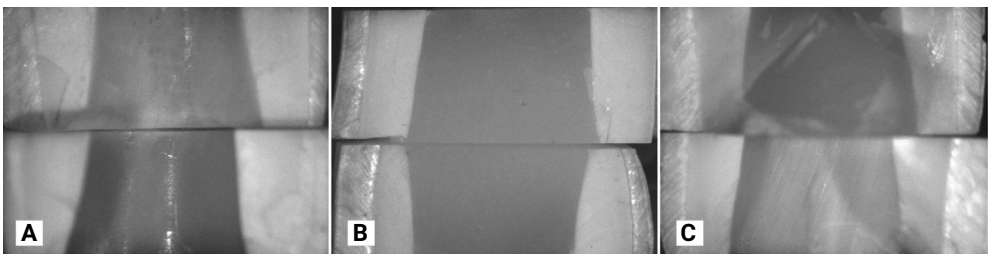

Figure 2. Failure modes obtained in this study: Adhesive (A), Cohesive (B), Mixed (C).

Statistical analysis

After confirming the parametric distribution of the errors, one-way ANOVA (for bottom/top hardness ratio) and two-way ANOVA (for bond strength) followed by Tukey posthoc tests were used to analyze the data ($p<0.05$). All statistical tests were performed using the GraphPad Prism 8 (GraphPad Software Inc, San Diego, California, USA).

Results

There were no statistically significant differences for the bottom/top hardness ratio ($p>0.05$). Comparisons among the groups are shown in Table 3.

Table 3. Means ± deviation from the bottom/top hardness ratio (B/T) and bond strength (Mpa) according to the resin composite and time-points tested.

Resin composites	B/T	Bond strength	
		24 hours	6 months
TNC	0.85 ± 0.16 a	10.57 ± 2.32 Aa	8.19 ± 1.63 Bb
TBF	0.90 ± 0.11 a	9.82 ± 1.99 Aa	10.38 ± 1.89 Aa
TNF	0.91 ± 0.24 a	2.15 ± 0.86 Bb	4.64 ± 155 Ac
PFS	0.95 ± 0.15 a	9.92 ± 2.08 Aa	6.92 ± 1.01 Bb
AFX	0.88 ± 0.09 a	10.63 ± 2.17 Aa	11.17 ± 2.65 Aa
XTB	0.90 ± .18 a	3.07 ± 0.83 Bb	3.88 ± 0.95 Ac

B/T: bottom/top hardness ratio. TNF: Tetric N-Ceram; TBF: Tetric N-Ceram Bulk fill; TNF: Tetric N-flow; PFS: Polofil Supra; AFX: Admira Fusion X-tra; XTB: X-tra Base. Different lowercase letters indicate statistically significant differences between the same time for different composites ($p < 0.05$). Different capital letters indicate statistically significant differences between the different times for the same composite.

There were statistically significant differences among resin composites ($p<0.05$) and time-points ($p<0.01$) for bond strength. Comparisons among the groups are shown in Table 3. At 24h, the resin composites TNC, PFS, TBF and AFX showed statistically higher bond strength than TNF and XTB. At six months, TBF and AFX provided the highest bond strength statistically, while TNC and PSF provided the lowest bond strength statistically. Considering the comparison between time-points, TNC and PFS showed statistically lower bond strength at six months, while TNF and XTB showed statistically higher bond strength at six months. TBF and AFX showed statistically similar bond strength between 24 h and six months.

Failure modes are shown in Figure 3. While adhesive failures were predominant for low-viscosity bulk-fill resin composites, other regular viscosity conventional and bulk-fill resin composites showed more mixed failures.

Discussion

The null hypothesis tested in this study - that there will be no statistically significant differences between the materials for both properties analyzed - was rejected. Although the B/T was not statistically affected by the different materials, statistically significant differences in bond strength were found among them.

As bottom/top hardness ratio of resin composites above 80% are adequate [12-15] all materials used in this study showed comparable polymerization between the bottom and top surfaces. Thus, even bulk-fill resin composites were inserted in the preparation in a single 4 mm thick increment, they were able to promote adequate polymerization in the depth region of the specimens. A factor that may have been crucial for this favorable result for bulk-fill composites is the quantity and type of monomers, their molecular weight, and the mobility of the tested resin composites. The greater translucency and similar refractive index of components of bulk-fill resin composites are often associated with increased light transmutation into the depth portion of the material, which might guarantee an adequate degree of conversion[5,16].

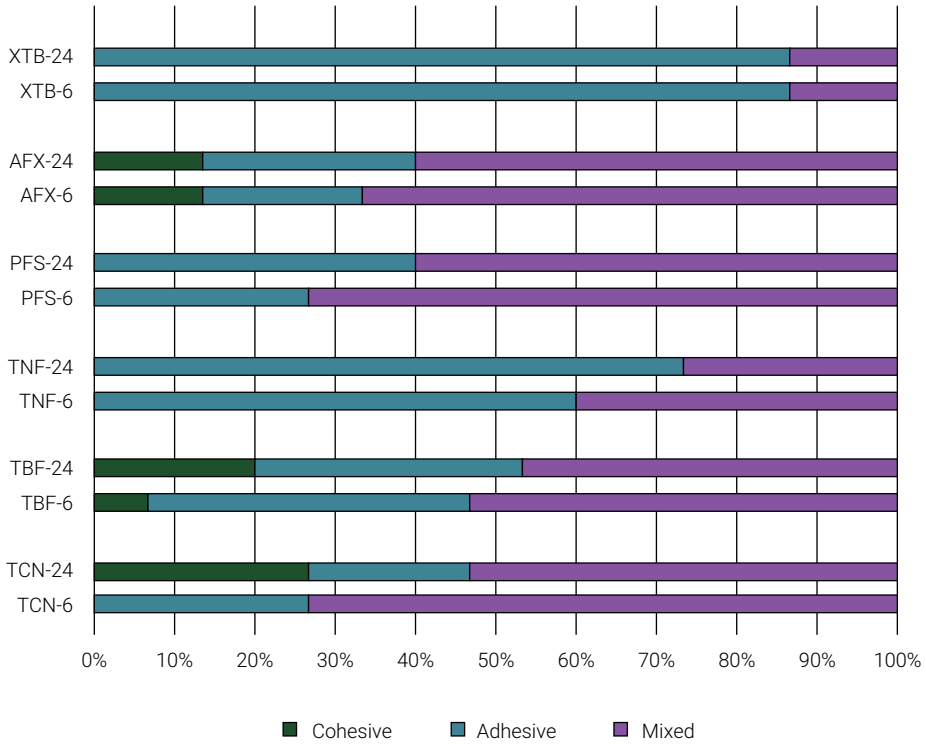

Figure 3. Distribution of the failure modes according to the resin composite and aging time analyzed. Column: aging time to perform the test (24 - 24h or 6 - 6 months) after specimen preparation. Lines: % of the failure modes. TNF: Tetric N-Ceram; TBF: Tetric N-Ceram Bulk fill; TNF: Tetric N-flow; PFS: Polofil Supra; AFX: Admira Fusion X-tra; XTB: X-tra Base.

Thus, the increments with thicknesses of up to 4 mm used in this research did not compromise the performance of the bulk-fill resin composites in the depth of cure compared to the conventional composites studied.

For bond strength, regular viscosity resin composites (either traditional or bulk-fill - TNC, TBF, PFS, and AFX) showed higher bond strength than low viscosity bulk-fill resin composites (TNF and XTB). Likely, low-viscosity bulk-fill resin composites have fewer filler particles, so bond strength decreased compared with resin composites containing more filler particles.

Conversely, only low viscosity bulk-fill resin composites provided higher bond strength at six months than 24 h. Less polymerization shrinkage stress was observed for a low viscosity bulk-fill resin composite than a traditional regular viscosity composite[10]. Also, low viscosity resin composites can dissipate easier polymerization stress due to a low elastic modulus than regular viscosity resin composites[5]. These findings may justify why only the low viscosity bulk-fill resin composites tested increased bond strength at six months of water storage. The higher elastic modulus of regular viscosity resin composites[4] may impair stress dissipation during polymerization. However, as a regular viscosity bulk-fill resin composite can show decreased polymerization contraction stress than a traditional resin composite[5],

only the dentin adhesive interface of preparations filled with traditional resin composites showed decreased bond strength at six months of water storage. The stress generated at the adhesive interface at the time of polymerization and aging can compromise the integrity of the dentin adhesive interface of preparations restored with conventional resin composites, resulting in loss of strength after six months[17].

This study used the push-out bond strength method to measure dentin bonding stability in high c-factor preparations. The bond strength of resin composites can also be analyzed using a microtensile test after filling Class I and Class II preparation, which requires cutting beams with diamond saws. Thus, external stress is transferred to the tooth/composite interface and may underestimate bond strength values. In contrast, the push-out method allows the measurement of bond strength without this external stress. In the push-out bond strength test, stress generated by polymerization is transferred directly to the adhesive interface, as the resin composite shrinks into the cavity[10].

Thus, the results obtained in this study state that regular-viscosity bulk-fill composite, in comparison with regular-viscosity and low-viscosity bulk-fill composite resins, may provide better clinical performance in terms of stability. However, more clinical trials need to be carried out to confirm this assumption.

Therefore, the bottom/top hardness ratio was not affected by the different materials tested. Only bulk-fill resin composites did not present dentin bond strength loss after six months of water storage. Only the low-viscosity bulk-fill resin composites were able to improve bond strength after aging.

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Data of availability

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

Author contribution

Maria Eduarda Lima do Nascimento Marinho: Drafting the work or revising it critically for important intellectual content; 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.

Rodolfo Xavier de Sousa Lima: Drafting the work or revising it critically for important intellectual content

Letícia Virgínia Freitas Chaves: Drafting the work or revising it critically for important intellectual content

Boniek Castillo Dutra Borges: Substantial contributions to the conception or design of the work; or the acquisition, analysis, or interpretation of data for the work; Final approval of the version to be published

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