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The impact of temperature on the shear bond strength of conventional multi-step and self-adhesive orthodontic adhesive systems: an in-vitro study

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Abstract

Background A recurring issue during orthodontic treatment is the detachment of brackets from the tooth surface, which proves problematic for both the patient and the orthodontist. This study aimed to evaluate the impact of various temperatures on the shear bond strength of metal molar tubes, using conventional multi-step and self-adhesive orthodontic adhesive systems.

Methods A total of 112 extracted human molars were randomly divided into eight groups ($n = 14$) for bonding tubes with two orthodontic adhesive systems (Transbond XT and GC Ortho Connect) at different temperatures: refrigeration temperature (4 °C), room temperature (20 °C), human body temperature (37 °C), and high temperature (55 °C). The shear bond strength (SBS) test was conducted using a universal testing machine set to a crosshead speed of 0.5 mm/min. The adhesive remnant index (ARI) was used to evaluate the amount of adhesive remnants on the molar surfaces. ARI scores were assessed under Carl Zeiss Stemi 2000-CS stereomicroscope with image recording camera AxioCam Mrc5 at $\times 10$ magnification. The data were analyzed using Student's t-test, parametric analysis of variance (ANOVA), the intra-class correlation coefficient (ICC), the Kruskal-Wallis test, and the chi-square test.

Results Higher mean SBS values were obtained with Transbond XT compared to GC Ortho Connect resin; however, the difference was not statistically significant ($p > 0.05$). The SBS results were lowest at 20 °C and highest at 55 °C in the Transbond XT group, and lowest at 37 °C and highest at 20 °C in the GC Ortho Connect group with no statistically significant difference ($p > 0.05$). The distribution of the ARI scores between the two materials showed a statistically significant difference ($p = 0.002$), with higher ARI scores found in the Transbond XT group.

Conclusions Pre-heating orthodontic adhesives prior to bonding does not affect the shear bond strength.

Keywords Shear bond strength, Orthodontic adhesives, Temperature

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Introduction

The prevalence of malocclusion among children and adolescents is remarkably high on a global scale, impacting at least one out of every two individuals or potentially more [1, 2]. For this reason, orthodontics is an inseparable part of today's dentistry world.

Currently, traditional braces continue to be the gold standard in orthodontic treatment due to their ability to achieve optimal teeth alignment [3]. Various protocols and materials are utilized to bond brackets to the tooth surface. However, the recurring issue during orthodontic treatment is bracket detachment from the tooth surface and it proves to be problematic for both the patient and the orthodontist [4]. Studies have shown that this problem occurs in 0.6–28.3% of all bonded brackets [5]. This issue has the potential to extend the overall treatment duration, increase chairside time and financial expenses, affect the compliance of a patient, while also compromising treatment outcomes and posing risks of enamel damage [6, 7].

Compared to brackets bonded on other teeth, bonded molar tubes were observed to have a higher failure rate (up to 28.8%) [8]. The failure of bonding can stem from patient-related aspects, such as variations in enamel composition or differences in masticatory forces, as well as procedural factors, such as difficulties in ensuring proper isolation during bonding, inadequate attachment adaptation to the tooth surface, or errors in etching and bonding techniques [7, 9].

Other factors can also influence the adhesive strength of orthodontic brackets. These factors include material-related aspects such as the type of etching material, type of bracket, bracket base design and size, as well as adhesives and bonding to restorative materials [10]. Additionally, tooth-related aspects such as fluorosis, bleaching and other miscellaneous factors play a role [11, 12]. Temperature is another significant element that can influence the adhesive properties of a tooth surface. Usually, adhesive materials are stored at room temperature to optimize their qualities, but it is also a common practice to refrigerate them to prolong their lifespan [13]. However, studies have shown that cooling composites before use can significantly lower the bond strength compared with those held at room temperature [14]. Contrary to that, the significantly enhanced repair strength was shown to be obtained after preheating these composites [15]. These results can be explained by the increase or decrease in adhesive viscosity after cooling or heating, changes in radical mobility, and degree of transformation [16–18].

New adhesive systems with different chemical compositions are continuously being introduced in orthodontic practice in the search for more efficient bonding. To simplify the application technique, manufacturers are

attempting to reduce the number of material components by introducing self-etching and self-adhesive bonding systems. Although initial studies on the influence of temperature on orthodontic bonding systems were conducted quite a long time ago [19], this topic has yet to be thoroughly investigated [13, 15, 20], particularly since heating composites is a technique commonly practiced in general dentistry and not in orthodontics. To the best of our knowledge, no research have compared the impact of temperature on the SBS of conventional multi-step systems, such as Transbond XT, and self-adhesive systems, such as GC Ortho Connect.

Hence, the aim of this study was to evaluate whether orthodontic bonding systems exposed to different temperatures affect the adhesion to enamel. Accordingly, the null hypothesis (H_0) states that exposing orthodontic bonding systems to different temperatures does not affect their adhesion to enamel.

Materials and methods

Preparation of the specimens

The present in-vitro study was performed at the Department of Orthodontics, Lithuanian University of Health Sciences. Bioethical approval was obtained from the Lithuanian University of Health Sciences Bioethical Committee (*No: 2024-BEC3-T-010*) and the methods were carried out in accordance with the relevant guidelines. Consent to participate was obtained from all of the participants in the study.

The sample size was determined using power analysis with G*Power statistical software (Version 3.1.9.7). The parameters adopted were as follows: a significant level of 5%, a power test of 80%, a standard deviation of 3.76, and the smallest effect of interest of 4. The following formula was implemented to calculate the sample size:

$$n = \frac{(\sigma_1^2 + \sigma_2^2) \times (Z(1 - \frac{\alpha}{2}) + Z(1 - \beta))^2}{\Delta^2}$$

when n - the minimum sample size for each sample; $Z(1 - \frac{\alpha}{2}) = 1.96$ and $Z(1 - \beta) = 0.842$ if $\alpha = 0.05$ and $\beta = 0.2$; σ_1 and σ_2 - standard deviations; Δ - the smallest clinically important difference.

The power analysis revealed that at least 14 specimens were needed in each group to determine reliable results.

Tooth inclusion criteria: Molars with intact buccal enamel surfaces recently extracted for periodontal purposes, with no decay, not damaged by fluorosis, without restorations, without endodontic treatment, and with no visible cracks. The extracted teeth were stored in a disinfectant (HistoPot, 4% formaldehyde) for 15 min, then washed under running water for one minute and kept in room temperature (22 °C) saline

(sodium chloride, Fresenius 0.9%). The isotonic solution was replaced daily to prevent bacterial proliferation. After applying the selection criteria, 112 permanent teeth from a sample of 253 were included in our study with any traces of blood and soft tissues meticulously eliminated, followed by a thorough rinse under a continuous flow of distilled water.

The teeth were randomly divided into eight groups ($n = 14$) for bonding with two available orthodontic adhesive systems at various temperatures: refrigeration temperature (4 °C), room temperature (20 °C), human body temperature (37 °C), and high temperature (55 °C).

The orthodontic resins selected for this study were Transbond XT® (3 M Unitek; St. Paul, MN, US – XT), a conventional multi-step adhesive system, and GC Ortho Connect (GC Orthodontics, Breckerfeld, Germany), a self-adhesive bonding system. Stainless steel GC Orthodontics metal molar tubes (GC Orthodontics, Breckerfeld, Germany) were chosen to bond the molar buccal surfaces.

As per the protocol, the buccal surface of the enamel was polished for 30 s using a rubber brush handpiece set at a low speed and non-fluoridated polishing paste. After that, the surface was rinsed with water for 30 s and compressed air was used to blow-dry it for 10 s.

Transbond XT group: The prepared area was etched with 37% phosphoric acid gel (Etch-37, Bisco, Schaumburg, IL, US) for 40 s, rinsed with water for 30 s, and dried with compressed air for 20 s until the enamel showed a faintly white appearance and became non-glossy. Using a micro-brush, the etched enamel surface was coated with a thin, even layer of binder resin (Transbond XT primer), and the air was blown until the binder became non-flowable.

GC ortho connect group: The prepared area was etched with 37% phosphoric acid gel (Etch-37, Bisco, Schaumburg, IL, US) for 40 s, rinsed with water for 30 s, and dried with compressed air for 20 s until the enamel showed a faintly white appearance and became non-glossy.

Utilization of the bonding composites occurred at four diverse temperature levels after being refrigerated in a refrigerator (Samsung RB38T602DSA/EF; Suwon-si, Gyeonggi-do, South Korea) or pre-heated in a multi block-heater (Thermo Fisher Scientific™; Waltham, Massachusetts, USA) for one hour before exposure: 5 °C, 20 °C, 37 °C and 55 °C (Fig. 1). The composite temperature was confirmed via a thermometer featuring a probe (ProfiCook DHT 1039 Stainless steel, Kempen, Nordrhein-Westfalen, Germany), which was immediately



Fig. 1 Pre-heating of the bonding composite in a multi block-heater

applied on the surface of the metal tube to avoid heat loss.

After that, the tubes were bonded to the center of the clinical crown with light-curing adhesives (four groups with the Transbond XT adhesive system and the other four with the GC Ortho Connect system) and pressed with a 100 g weight on the buccal tooth surface using a Hollenback carver. The excess adhesive was removed using a dental scaler. Properly placed molar tubes were cured with a polymerization lamp (Foshan, Guangdong, China) for 40 s (20 s from the occlusal aspect and 10 s medially and distally to the molar tube), and the light source was positioned one millimeter from the tube surface. The bonding procedure was conducted by one person to maintain accuracy.

The prepared samples were embedded in a self-cured acrylic block and maintained in saline for 24 h before the shear bond strength test was performed.

Shear bond strength test

The SBS test was conducted using a universal testing machine (TINIUS OLSEN H10KT, Horsham, US). The prepared samples were anchored to the metal base plate to prevent them from moving while the test was being carried out. A cap screw was fixed in the movable cross-head of the testing machine and the leading edge of its plane, which was prepared exactly for this purpose, was positioned vertically to aim at enamel-composite conjunction (Fig. 2). A force was then exerted to the flat interface, directed occlusal-apically. This force is intended to simulate the mastication force. A speed of 0.5 mm/min was set for the crosshead. The maximum force required

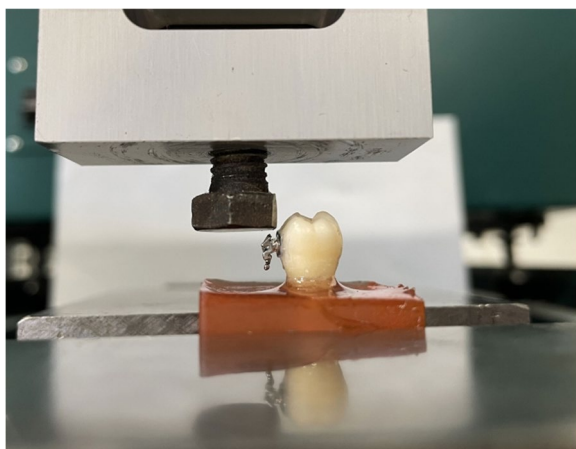


Fig. 2 The prepared sample fixed in the universal testing machine

to detach the molar tubes was measured in newtons, and the SBS was calculated by dividing the force values by the tube base area in square millimeters (1 MPa = 1 N/mm²). We calculated the area of each tube to be approximately 18 mm².

Failure mode analysis

Post-debonding, the amount of adhesive remnants was evaluated by using the ARI on all the molar surfaces. ARI scores were examined under Carl Zeiss Stemi 2000-CS stereomicroscope with image recording camera AxioCam Mrc5 at ×10 magnification. Teeth were scored according to the ARI as follows: 0 = no adhesive remaining on the tooth; 1 = less than 50% of the adhesive remaining on the tooth; 2 = more than 50% of the adhesive remaining on the tooth; 3 = all adhesive remaining on the tooth with a distinct impression of the tube base. To evaluate the reproducibility of the ARI measurements made by two investigators (GK and RV), the intra-class correlation coefficient (ICC) was calculated. The ICC indicated a high agreement between the two investigators (0.98; 95% CI: 0.97; 0.99, *p* < 0.001).

Statistical analysis

All the statistical analyses were performed using IBM SPSS Statistics 29.0.0.0. Normality was determined via the Kolmogorov-Smirnov test. SBS data were analyzed by Student’s t-test, and parametric analysis of variance (ANOVA). The Kruskal-Wallis test was used to assess the distribution of the ARI scores across the groups. The chi-square test was employed to compare the incidence of enamel fractures between the materials and to determine whether the differences observed were statistically significant. The results were considered statistically significant if the *p*-value was < 0.05.

Results

Shear bond strength analysis

The outcomes of the SBS analysis are presented in Table 1. The mean SBS measurements were 11.76 MPa for Transbond XT and 11.37 MPa for GC Ortho Connect. Higher mean SBS values were obtained with Transbond XT compared with GC Ortho Connect resin; however; based on the parametric Student’s t-test for two independent samples, it was found that the shear bond strength, considering the materials, showed no statistically significant difference (*p* > 0.05).

In the Transbond XT group, the lowest SBS values were recorded at an adhesive temperature of 20 °C, and the highest values - at 55 °C, whereas in the GC Ortho Connect group, the SBS results were found to be the lowest at an adhesive temperature of 37 °C and the highest - at 20 °C. Nonetheless, based on the parametric analysis of variance (ANOVA), the shear bond strength, with respect to different temperature groups, was no significantly different (*p* > 0.05).

Adhesive remnant index

The remaining adhesive on the enamel surface was assessed using the ARI, as shown in Fig. 3. When comparing the two materials used to bond the tubes to the tooth surface, a statistically significant difference was found (*p* = 0.002), with higher ARI scores observed in the Transbond XT group. The distribution of the ARI across

Table 1 Shear bond strength (MPa) of two types of orthodontic resins at multiple temperatures. SD = standard deviation; Min = minimum; Max = maximum

Temperature	Transbond XT			GC Ortho Connect			<i>p</i> -value
	Mean (SD)	Min	Max	Mean (SD)	Min	Max	
4 °C	11.15 (2.01)	7.73	14.21	11.63 (2.83)	5.27	16.18	0.624
20 °C	10.27 (3.35)	5.48	17.04	12.27 (3.79)	6.34	17.66	0.123
37 °C	12.46 (4.62)	5.57	21.51	10.40 (3.26)	5.40	15.98	0.270
55 °C	13.11 (3.79)	7.07	19.38	11.28 (3.71)	7.14	18.13	0.198
<i>p</i> -value	0.209			0.570			

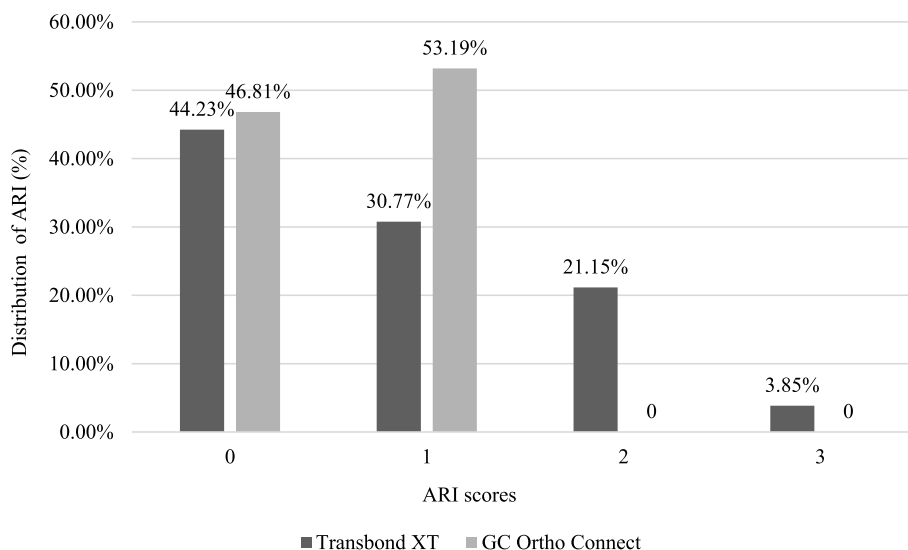


Fig. 3 The distribution of the adhesive remnant index (ARI) scores between the resins

the different temperatures showed no significant difference ($p > 0.05$). Six cases of enamel fracture were detected and included in this study (5.7% of the samples). Five cases were observed in the GC Ortho Connect group, distributed as follows: three cases in the 20 °C subgroup, one case in the 4 °C subgroup, and one case in the 55 °C subgroup. The remaining case occurred in the Transbond XT group at 37 °C. A statistically significant difference was not observed in the incidence of enamel fractures between Transbond XT and GC Ortho Connect ($p > 0.05$) when compared using the chi-square test.

Discussion

The findings of this study support the null hypothesis (H_0), indicating that exposing orthodontic bonding systems to different temperatures does not significantly affect their adhesion to enamel. This suggests that temperature variations within the tested range have no meaningful impact on the bonding performance of these systems.

The minimum shear bond strength required for orthodontic bracket bonding is not universally defined and can differ based on various studies and specific clinical needs. As per Reynolds, the minimum SBS required in orthodontic treatment is between 5.9 and 7.8 MPa [21]. An ideal orthodontic biomaterial should provide sufficient adhesion to withstand chewing loads of 5–10 MPa, while ensuring that the adhesion is not excessively strong to avoid damage to the enamel during debonding (40–50 MPa) [22]. The results of the present study demonstrated that the two adhesive systems maintained adequate bond strength within the range of 5.27–21.51 MPa

when applied at different temperatures. However, there were seven cases where SBS values were lower than the minimum required SBS, indicating bond failure and insufficient attachment of the molar tubes to the teeth; therefore, these cases were excluded from this study.

The mean SBS values of Transbond XT were greater than those of the GC Ortho Connect resin; however, the difference was not significant. Iglesias et al. also did not find a statistically significant difference between conventional and self-adhesive systems [23]. In contrast, other studies have shown significantly lower SBS of the self-adhesive resins in comparison to the conventional etch-and-rinse adhesive system [24, 25].

Some studies have indicated that temperature has a minimal impact on shear bond strength [15, 26], while other studies have demonstrated that temperature variations can indeed affect SBS values and potentially influence the adhesion of orthodontic biomaterials to enamel [13, 20, 27]. In the present investigation, the two adhesive systems yielded different outcomes, although the results were not statistically significant. In the Transbond XT group, the minimum bond strength values occurred at 20 °C. However, quite a few studies have reported the lowest SBS values at 4 °C, using either dental composites for restorations [27, 28] or adhesive systems in orthodontics [13]. Lower temperatures typically result in higher viscosity of dental adhesives, making them thicker and less capable of penetrating into the irregularities of the tooth surface and bracket base mesh, thereby creating worse adaptation and weaker composite-tooth and composite-bracket interactions [29, 30]. A decrease in temperature also results in decreased mobility of monomer

molecules within the resin matrix of the resin-based composite and more constrained radical formation, which ultimately leads to weaker bonding [31].

The maximum SBS in the Transbond XT group was recorded at 55 °C. This outcome aligns with the findings of Akarsu et al. [27], who reported that adhesive systems obtained the highest SBS values when heated to 55 °C. Further studies revealed that adhesives pre-heated to 60 °C presented the highest SBS values [15, 20]. Heating the composite can enhance the polymerization reaction [32] and increase monomer conversion, leading to improved physical characteristics of the composite: higher surface hardness, greater flexural strength, enhanced mechanical strength and wear resistance [18, 33]. Raising the polymerization temperature of a resin composite lowers the viscosity of the material, leading to increased flow due to the enhanced movement of monomer molecules [17].

In the GC Ortho Connect group, the lowest bond strength values were obtained at 37 °C. This orthodontic adhesive system differs from Transbond XT because it is used without a separate binder or primer resin, which could have influenced the study's results by increasing the risk of microleakage at the enamel-adhesive interface [34]. Transbond XT and GC Ortho Connect orthodontic adhesives also differ in their chemical compositions, which can affect their performance and properties. Both adhesive systems are resin-based composite materials that normally contain a combination of Bis-GMA (bisphenol A-glycidyl methacrylate) and TEGDMA (triethylene glycol dimethacrylate), as well as filler particles such as silica [35]. However, GC Ortho Connect also incorporates a phosphoric ester monomer and 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which ensures a stable bond, eliminating the need for a separate primer.

Therefore, these two adhesive systems could have different reactions to temperature changes, affecting the film thickness and viscosity [36]. Pre-heating the GC Ortho Connect adhesive decreased its viscosity and made it overly fluid, resulting in sliding of the tubes and making it difficult to control during application. Additionally, after tube seating, the material tended to spread more readily along the tube borders. In fact, the downward flow of material due to gravity is a major drawback for bonding of tubes as it may lead to inadequate coverage, an uneven polymer network, and weaker adaptation of the composite to the tube base [24]. Hence, the highest SBS was obtained at 20 °C. This result is in agreement with Sharafeddin et al., who reported that pre-heating the materials had no significant effect on shear bond strength and that the highest values were obtained at room temperature [28].

In regard to bracket debonding at the end of orthodontic treatment, there is an increased risk of enamel fracture or even tear-out [37]. In the literature, a variety of risk factors are listed, including the type of instrument and force used for bracket debonding, the type of material and protocol used to bond brackets, the bond strength between the enamel and adhesive, and the type of bracket used for treatment [38–41]. In the present study, six cases of enamel fracture were observed, with no statistically significant difference when comparing either the materials or the temperatures. Rix et al. suggested that the increased number of enamel cracks may be due to tooth extraction forces and could be lower when tested in vivo [42].

The amount of adhesive remnant on the tooth surface after bracket debonding depends on several factors, including bracket base design and the qualities of the adhesive type [43]. In this study, the bracket base design should not have influenced the adhesive remnant index score, because identical brackets were used for every specimen tested. Usually, three types of adhesive systems are used to bond brackets to teeth: conventional multi-step, self-etching, and self-adhesive. In the present study, conventional multi-step (Transbond XT) and self-adhesive (GC Ortho Connect) resins were used. A comparison of these materials has shown a statistically significant difference, with higher ARI scores observed in the Transbond XT group. Bracket failure typically occurs at the weakest link in the adhesive junction. The amount of adhesive remaining on the tooth surface after debonding can be explained by the bond failure mode: adhesive-enamel, adhesive-bracket, and cohesive. Bracket bonding with the conventional multi-step adhesive used in this study tends to show cohesive or adhesive-bracket bond failure modes, indicating that a greater amount of material remains on the tooth surface. These results are in accordance with the literature [23, 25]. In contrast, bonding with self-adhesive resin results in bond failure at the adhesive-enamel interface, showcasing most of the adhesive residues on brackets rather than on the tooth surface. Brackets bonded with self-adhesive material tend to have lower SBS values, and a weaker bond between enamel and resin is observed, leading to bond failure at the adhesive-enamel interface [25]. Cohesive or adhesive-bracket bond failure is generally considered 'safer' rather than adhesive-enamel debonding because it leaves the enamel surface relatively intact; however, the removal of the residual adhesive increases the possibility of damaging the enamel surface during the cleaning process [25, 44]. Nevertheless, in more than 40% of the cases, adhesive-enamel bond failure was observed with both adhesive systems, which corresponds to the results of Lobato et al., where ARI scores of 0 or 1 were predominant [15].

While in Borges's study, which compared composite restoratives to conventional orthodontic adhesives, the entire composite stayed on the tooth surface in most groups [26]. Bishara et al. and Iglesias et al. also reported that, for the Transbond XT group, the majority of the adhesive remained on the tooth after debonding [23, 25]. On the other hand, less adhesive residue on the enamel surface after bracket debonding may be desirable in clinical practice as it reduces the chairside time. However, enamel fractures can occur during the debonding procedure [44].

In this study, the distribution of ARI across different temperatures showed no significant difference. These findings are similar to those of other studies, which also revealed no significant differences in the ARI at low and high temperatures [13, 15, 20].

It is important to note that it might be challenging to compare the results of different studies because a number of variables could account for discrepancies in the results. For example, the type of teeth selected for the study, the use of orthodontic adhesive systems as well as brackets/tubes from the same manufacturer, the application of identical temperatures, and the operator's influence all highlight the need for standardizing the methodology to enable more efficient comparison of results [45].

Conclusion

There was no statistically significant difference in the shear bond strength of the conventional multi-step and self-adhesive orthodontic adhesive systems at the four different temperatures. Regardless of whether a separate binder is applied when bonding tubes to the enamel, good bond strength is maintained. Significantly higher adhesive remnant index scores were recorded when the tubes were bonded with the conventional orthodontic bonding system.

Recommendations for clinical applications

- Preheating is not necessary: Preheating orthodontic adhesives does not significantly affect SBS and can be omitted to simplify procedures, though it may aid in handling by reducing viscosity.
- Store adhesives at room temperature for consistent performance. If refrigerated, ensure they return to room temperature before use to avoid increased viscosity and reduced flow.
- Due to the higher failure rates of molar tubes, clinicians should prioritize precise adhesive placement,

proper isolation, and appropriate adhesive selection to minimize bond failure.

- Both adhesives tested provided clinically acceptable SBS within standard ranges, but care should be taken to avoid conditions that may lead to suboptimal bond strength.
- Additional clinical studies are needed to validate these findings in vivo and explore temperature effects on other adhesive systems.

Abbreviations

SBS	Shear bond strength
ARI	Adhesive remnant index
SD	Standard deviation
Min	Minimum
Max	Maximum

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Authors' contributions

G.K. and R.V. substantially contributed to the conceptualization and design of the work, the acquisition, analysis, and interpretation of data, and drafted the manuscript. D.S. substantially contributed to the conceptualization and design of the work, the analysis and interpretation of data, and the substantive revision of the manuscript. K.L. substantially contributed to the interpretation of data and the substantive revision of the manuscript. B.P. substantially contributed to the conceptualization and design of the work. A.J. substantially contributed to the acquisition and interpretation of data and the substantive revision of the manuscript. All authors read and approved the final manuscript.

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Data availability

The data supporting the conclusions of this study are confirmed by the authors to be accessible upon request.

Declarations

Ethics approval and consent to participate

Bioethical approval was obtained from the Lithuanian University of Health Sciences Bioethical Committee (No: 2024-BEC3-T-010) and the methods were carried out in accordance with the relevant guidelines. Informed consent to participate was obtained from all of the participants in the study.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests.

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