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Original Research

Effects of precuring heating modalities on the shear bond strength and adhesive remnant index of orthodontic adhesive

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Aim: This study aimed to investigate precuring adhesive and bracket heating\cooling protocol on shear bond strength (SBS) and adhesive remnant index (ARI). **Methods:** 70 newly extracted lower premolars categorized randomly into 7 groups (n = 10 (ten teeth each)) according to adhesive and/or brackets temperatures. Pre-curing, the 3M Transbond Plus™ XT adhesive temperature was adjusted to 5, 22, 40, 70°C, while the brackets were stored at 5, 55°C before bonding. Also, adhesive and brackets were heated by tooth dryer before curing representing the seventh group. The samples were stored in deionized water for 24h at 37°C in dark incubator before debonding by universal testing machine. Analysis of variance (ANOVA) and post Hoc Tukey statistical tests were performed for SBS analysis, while Kruskal Wallis test analyzed ARI data (α=0.05). **Results:** Significant differences were found among the groups regarding SBS and ARI where p = 0.00, 0.024 respectively. SBS significantly increased when the adhesive temperature modified to 5°C and 70°C and brackets temperature adjusted to 5°C and 55°C. Also, heating the adhesive and brackets by tooth dryer generated peak SBS. While, heating the adhesive to 40°C declined SBS significantly. **Conclusions:** Lowering the temperature of the adhesive and/or brackets to 5°C and elevation their temperature above 55°C pre-curing has favorable effects on SBS. Storing the adhesive at 40°C before bonding showed reductions in SBS. Also, heating both the adhesive and brackets pre-curing formed all-out SBS with reduced ARI.

Keywords: Dental Cements. Heating. Cold temperature. Shear strength.

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Introduction

In orthodontics, polymeric adhesive resin is employed as dental bonding system that secure an intimate and resilient adhesion between the teeth buccal surface and brackets base[1]. The adequate bond strength between teeth enamel and brackets (which range between 6-8 MPa) is the most important goal of ant orthodontic adhesive systems in order to resist the dislodgment effect of mastication and orthodontic forces and insure effectiveness and efficiency with reduce cost of the treatment with fixed orthodontic appliances[2,3].

The success of orthodontist treatments requires an adhesive that is easy to manipulate with proper fluidity to keep the bracket over the tooth surface while it is being lightly-cured[2]. The bond strength is affected by many factors such as the adhesive quality of the bonding material, the adhesion at the tooth – composite interface, the composite age, the viscosity of the adhesive and storage media. Also, the shape and size of the bracket base besides enamel etching technique and the environmental temperature and humidity[4]. During polymerization, the environmental temperature plays an important role in improving the reaction process by increase the activations of the functional groups within the chemical composition of the un-cured monomer. This finally could improve the mechanical properties of the produced polymer. During bonding of the orthodontic brackets, a sufficient amount of un-cured adhesive is implicated on the base of the bracket and forced toward the tooth surface before starting the polymerization process[5].

Storage temperatures of the adhesive before using is controversy between clinicians. As most of the manufacturers recommend the use of the adhesive material at room temperature, the clinician tend to keep it at the refrigerator in order to extend its shelf time. Some of them tend to use the adhesive immediately deprived of reaching room temperature which has been claimed to affect the mechanical properties of the adhesive material[6,7].

On the other hand, preheating of the resin composite prior to light activation showed a higher polymerization rate and improve the degree of conversion[8 9].

Most of the published studies[10,11] that had been conducted on the effects of adhesive thermal changes on the SBS of orthodontic adhesive focused on cooling or heating the adhesive material. Another possible option could be heating/cooling of the brackets before initiation of the polymerization by light cure. As the thickness of the orthodontic adhesive underneath is about (0.23) mm[12] which could easily be affected by the temperature of the underlining bracket base. Furtherly, heating of the brackets and adhesive immediately before curing could be a possible option.

The aims of the current study were to find out the effects of changing adhesive temperature and/or bracket base surface temperature on the SBS and ARI to reach the best pre-curing heating- cooling protocol of orthodontic adhesive and bracket base before bracket bonding regarding SBS and ARI. The Null hypothesis implies that changing of the adhesive and/or tooth bracket base temperature pre-curing has no effect on the SBS and ARI.

Method

Search strategy

This cross-sectional study was performed in vitro at the Collage of Dentistry/University of Mosul extended from January 2024 to April 2024. The study protocol was reviewed and agreed by the Research Ethics Committee (REC) of the Collage of Dentistry/University of Mosul.

The sample size calculation was created according to the results of the Power Analysis of the G*Power Software. As the estimated sample size of each proposed group for the current study was 10 samples to qualify of 80% power and a 5% level of significance[13,14]. Consequently, 70 newly extracted lower premolars were clustered for this study. The teeth were collected from orthodontic centers at the dental school and all the teeth were extracted for orthodontic aims. The inclusion criteria of the selected teeth were: Absence of cracks which was checked by Stereomicroscope at 10 magnification power, no-carious nor filled premolars teeth on all enamel surfaces[15.] The teeth were washed with distilled water and stored in 0.05 chloramine T solution for fourteen days, to confirm disinfection before starting the procedure. The premolars were mounted in cylindrical self-cured acrylic blocks. The crowns were adjusted to be exposed to shear bond test. Dental surveyor was performed for adapting the buccal surface of the teeth, parallel to the shear force applied during test. The teeth were cleaned and polished with non-fluoridated pumice for 15 sec using lower speed hand piece with rubber cup and stored in distilled water for further procedures.

The 70 lower premolar teeth blocks were randomly clustered into seven groups (n=10 blocks each) based on the heating protocol of the current study, the temperature of the adhesive and\or the teeth surface were set as follows:

1. Group 1: Adhesive 22°C group (A22°C); the adhesives and the brackets were stored at room temperature before brackets bonding procedure.

2. Group 2: Adhesive 40°C group (A40°C); the adhesive was stored in Vokodak Dental Heat Composite Warmer (model no, VK-2301, Foshan, China), which was adjusted to 40 °C and the adhesive was used after blue bulb of the heater was on to ensure reaching the target temperature of the adhesive inside the tube. While the brackets were stored at room temperature (22°C).

3. Group 3: Adhesive 70°C group (A70°C); The adhesive was stored at composite heater adjusted to 70 °C. The adhesive was used after a yellow lamp of the heater was switched on that indicated complete heating of the adhesive to the target temperature. The brackets were stored at room temperature (22°C).

4. Group 4: Adhesive 5°C group (A5°C); The adhesive was stored in Refrigerator (Arcelik Refrigerator 70505 EI, Istanbul, Turkey) at 5°C for 24 hours before starting the procedure. The brackets were stored at room temperature (22°C).

5. Group 5: brackets cooling 5 (BBS5 °C) group; the brackets were stored in Refrigerator at 5°C for 24 hours before starting the bonding procedure. While

the adhesive was stored at room temperature. Bracket base surface temperature was measured by Würth Infrared laser thermometer (Künzelsau-Gaisbach, Deutschland) before curing.

6. Group 6: bracket base surface 55 (BBS 55°C) group; the brackets were stored in warm water adjusted to be 55° C for 180 sec, While the adhesive was stored at room temperature.

7. Group 7 : (HA) group; the tooth, bracket and adhesive complex was heated before and during curing, the NOLA Warm Air Tooth Dryer (Gaithersburg, MD 20879, USA) was applied perpendicular to tooth surface and bracket in order to heat the underneath adhesive for 10 sec before start curing (the tooth surface temperature was measured close to the bracket by the infra-red thermometer, while the adhesive temperature underneath the bracket was measure by Extech Type K thermometer, Bunker Court Vernon Hills, IL, USA). The sensor end of the K thermometer was inserted in a small notch which was prepared on the peripheral part of the bracket base to insure contact with the adhesive. The hot air source was on for additional 10 sec during curing.

The hot air source was adjusted to 30 mm away from the tooth surface at right angle to bracket. Adhesive and water temperature in the composite heater was controlled by LABWORLD Glass Mercury Thermometer (Union Road, London UK), while bracket surface temperature was measured using Würth Infrared laser thermometer (Künzelsau-Gaisbach, Deutschland) before curing.

Bonding procedure

A sufficient amount of 37% phosphoric acid gel (Biodinamica, Brazil) was applied on the buccal surface of the teeth for 30 seconds. Then, the teeth were thoroughly rinsed by triple syringe distilled water for 15 seconds. The tooth surface was dried using oil and water free air syringe of the dental unit for 30 sec, until the tooth surface appeared white and chalky. The Transbond XT Primer (3M Unitek, Monrovia, CA, USA) was applied over the etched surface using small brush, followed by air jet, and curing for 10 seconds. A sufficient quantity of orthodontic adhesive (3M Transbond™ XT, USA) was applied over the base on the brackets and pushed on the tooth surface with a constant load of 150 g load to gain a uniform thickness of the adhesive. The extra adhesive was removed from bracket base periphery by a sharp probe, the curing protocol was performed for 20 sec, 10 seconds on gingival and occlusal aspects respectively, by a LED light source unit (Woodpecker LED ORTHO curing light, China) at constant distance (2 mm) from the bracket margins to the light source using fully charged cordless led , LED curing light with intensity 1550 (mW/cm2) which was restrained by LM1 DTE Light Curing Lamp Power Meter (Foshan, China), and standardized continuously throughout the study, to confirm consistent light intensity. Roth 0.022-inch slot size IOS Stainless steel metal brackets for lower premolars were used in all groups. The brackets surface area was (10,5 mm^2). A new adhesive tube was used for each group (from group 1 to 5) to avoid any possible deformities of the chemical structure of the adhesive after exposure to heating and cooling. All the bonding procedure was done at the same day at room temperature 22°C which was controlled by air condition and internal thermometer. The bonded samples were preserved in dark container filled with deionized water at 37°C for 24 hours before subsequent shearing tests[13,15.]

A universal testing machine (Gester Instrument Co., Fujian, China) was used for SBS test. The crosshead speed was adjusted at 0.1 mm/min. for all samples, as the chisel Edge blade was adapted at the adhesion interface between the bracket and enamel, in this manner, the bracket base was parallel to the shear loading path, and producing de-bonding force at the bracket-enamel interface. The de-bonding rod was adjusted after each shearing test. The SBS records were uttered in MPa by dividing the force results (in Newton) on the surface area of the bracket base (in mm^2). For ARI analysis, immediately after brackets de-bonding, the enamel surface at the bracket adhesion side was observed under 20x magnification using SZ61 Olympus stereo microscopes (Shinjukuku, Tokyo, Japan), to decide the extent of remaining adhesive on the tooth enamel surface by a blinded examiner agreeing to the categories of adhesive remnant index (ARI), as follow:

Grade 0 where no adhesive remaining on the enamel surface; Grade 1 where less than half percentage (50%) of the adhesive remaining on the enamel surface; Grade 2 where more than half percentage (50%) of the adhesive remaining on the enamel surface; and finally Grade 3 where the entire adhesive remaining on the enamel surface with display of bracket base impression[16.]

To overcome any probable errors during the ARI recording, three groups of the total samples (Groups 3, 5 and 7) were nominated by simple random method to be counted over by the same operator three weeks after the primary recording. Precisely the identical scores were attained in the subsequent recording session. Later, the de-bonded enamel surface was inspected for any cracks on the enamel surface using a stereomicroscope.

The Shapiro-Wilk test was performed to validate the normal distribution of the SBS data outcomes. Thus, the parametric tests (one-way ANOVA and post-hoc Tukey HSD tests) were utilizes for data examination. While, Kruskal- Wallis test was performed for analysis of ARI outcomes, as ARI data did not demonstrated a normal distribution. Furtherly, Mann-Whitney test was performed to compete the statistical differences between the ARI groups. Significance level for entirely statistical analysis was set at $\alpha=0.05$, performing SPSS software version 26.

Results

The descriptive outcomes (mean and standard deviation) of the SBS, Bracket base surface temperature and adhesive temperature at curing were listed in the table 1. Shapiro-Wilk test discovered a normal distribution of the data for all tested groups as shown in table 1. Agreeing to one-way ANOVA, there was significant differences among all tested groups where ($p < 0.05$).

Table 1. The mean and standard deviations of Shear bond strength, Bracket Base surface temperature and adhesive temperature with different heating protocol.

Group number	Groups description	Mean/Std. Deviation MPa	F	Sig.**
1	A22°C	15.6223 ± 1.014 AC	15.057	.000
2	A40°C	11.5736±1.49 A		
3	A70°C	18.8663± 1.9 BC		
4	A5°C	19.2215± 1.99 BC		
5	BBS5°C	21.53± 1.261 CD		
6	BBS55°C	18.39± 2.741 BC		
7	HA	23.1606± 2.189 D		

**ANOVA test.
Groups marked with different letters (upper case) validate significantly different outcomes of the Tukey post-hoc HSD test, where $p < (0.05)$.

The Kruskal-Wallis test findings revealed that ARI scores were significantly different ($p = 0.024$) between all groups as shown in table 2, where ($p < 0.05$). The second group (A40°C) expressed high ratio of adhesive remain in the de-bonded teeth surface after debonding. The Mann-Whitney test showed no significant differences between some groups which was presented in different letters in the same row as shown in table 2.

Table 2. Adhesive remnant index (ARI) scores' percentage for all groups with different heating protocol.

Group number	Group	0	1	2	3	*p** value
1	A22°C abde	0%	40%	60%	0%	0.024
2	A40°C abc	0	20%	40%	40%	
3	A70°C a	20%	20%	60%	0%	
4	A5°C a	20%	60%	20%	0%	
5	BBS5°C abce	20%	20%	0%	60%	
6	BBS55°C abde	40%	40%	20%	0%	
7	HA abde	2%	8%	0%	0%	

*The data were analyzed using the Kruskal-Wallis test. Letters in the same row are not significantly different at $P < 0.05$ according to The Mann-Whitney test.

Discussion

Adequate polymerization is an important factor to attain appropriate degree of conversion and good physical and mechanical characteristics of resin based composite material. Additionally Appropriate enamel surface and bracket base is a critical factor in an effective orthodontic treatment, as the acceptable strength of the tooth bracket shear bond ranged between 6-8 MPa[1,6]. 3M Transbond™ XT adhesive with medium viscosity was used in this study as it considered the golden standard adhesive with a homogenous chemical structure.

The null hypothesis was not accepted regarding the SBS and ARI.

Heating modalities selection depended on most frequent situation faced during orthodontic practice which is refrigeration temperature (5°C), as most of the orthodontists store the adhesives in refrigerator to increase their shelf life, while others store at room temperature (22°C). the possible available option is heating the adhesive by the composite heater (that used for Dental composite) with its 2 temperature options (40, 70°C) precuring. the other possible option is to cool or heat the bracket base as a substrate for the polymerization process for different possible temperatures, as well as investigating the effect of heating of the tooth, bracket and adhesive as a one unit before curing as a final possible option employing the tooth dryer on the SBS and ARI of the orthodontic adhesive.

The selected temperatures of 5 °C for cooling and 55 °C for heating the bracket base surface before bonding represented the minimum and maximum degree that human can withstand without a harmful effect to dental tissue and the pulp. Additionally, these selected temperatures conform with the ISO TS 11,405 technical specification for assessment the adhesion to the enamel surface[14,17]. These degrees could cover the clinical implication of this study.

A coordination was observed between the results during heating or cooling of the adhesive and bracket base surface, this could be attributed to the thin film of the adhesive under the brackets[12.] This thin film of the orthodontic adhesive can be affected faster with the bracket base temperature, and decreasing adhesive viscosity inducing simple spread out the adhesive on the etched tooth surface. The thin film of the orthodontic adhesive could be contributed to the non-significant relation between groups that are cooled or heated to related temperature of the adhesive and the bracket base surface as shown in table 1.

The scientific interpretation of the improving the SBS after heating of the bracket base and the adhesive could be attributed to the effect of heating on the degree of conversion of the adhesive, as the dental composites degree of conversion is incomplete at room temperature, and raising the temperature of the adhesive to certain limit had been shown to reduce its viscosity resulting in higher polymerization and developed degree of conversion, as it increased radicular mobility[2,] which in turn, could be related to improvement in the free volume accrual of the monomer that enhanced the mobility of the trapped free radicals combined by decreasing adhesive viscosity which, finally improved the confrontation incidence of the free radicals with the unreacted monomer active groups[18]. However, in the current study the temperature was raised under the TG temperature of the orthodontic adhesive.

As most of the studies[10,11] that have evaluated the SBS of orthodontic bracket bonding concentrated on manipulating the temperature of the adhesive either by elevating or demoting, changing of the bracket base surface temperature had not received the same attention as a possible option that could be applicable in the dental practice. Elevating or decreasing bracket base surface temperature can affect the adhesive polymerization and SBS due to thermal exchange of the thin adhesive film underneath the brackets[5,19-21].

The current study involved raising the adhesive and the bracket base surface temperature (above 55 °C) as well using a tooth dryer precuring. A maximum increased in SBS has been gained which may imply an enhanced degree of conversion and improved polymerization[22.] Also, heating decreased the viscosity of the adhesive leading to a higher flow of the material on the bracket base mesh and the microporosity on the etched tooth surface creating a stronger bond between bracket and adhesive[18]. However; according to the current study outcomes, raising the temperature of both adhesive and tooth surface recorded the highest SBS value indicating a better bonding, as heating could enhance the mobility of the radicals of the adhesive and improve their reaction ability. Our findings come in agreement with those of Freidman[10] who recorded an elevated degree of conversion when the composite resin heated to 54- 60°C.

Maier et al.[23] support Eliades and Caputo[24] opinion, in that pre-heating of the composite resin above 54 °C might increase segmental mobility of the C = C double bonds of the superficial layer and of the radicals produced, that support the co polymerization with the neighboring chain.

On the other hand, this study revealed that lowering the temperature of bracket base surface or the adhesive, increased the SBS, and this could be credited to the increase of the hydrogen bonds strength between the radicals that is affected by the polymer temperature or the underneath substrate, which in turn increased in number and durability as the temperature decreases[13]. Nevertheless, decreasing the temperature has an impact effect on the viscosity of the adhesive. This finding come in agreement with that of de Araujo et al.[25] who found a negative correlation between the viscosity of the adhesive and it is degree of conversion. The viscosity of the orthodontic composite is related to the filler content and resin type used in their structure[22]. However, in this study one type of orthodontic adhesive was used to overcome the differences in the resin type and percentage of filler content. This correlation between the filler and resin type highly effects the monomer conversion as they can bind to the monomer's radical mobility, thus, propagate the polymerization of the adhesive[14,26] .Cooling of the adhesive reduces the viscosity of resins and enhances monomeric mobility and better distribution of the free radicals inside the adhesive, that can improve polymerization. Also, it was reported that low viscous composite has a high penetration ability to enamel porosity that is induced by acid etched directly without the need of intermediated bond[27.]

Moreover, the adhesive, cohesive and Van der Waals forces are increasing with decreasing temperature as the two latter depends on hydrogen bonds. These outcomes disagree with Reis et al.[28] who found no effect of lowering the temperature on SBS. Also, it comes in contrast with that of Akarsu et al.[11] who reported a decrease of SBS when the orthodontic adhesive cooled to 5°C.

According to the outcomes of this study, one of the surprising findings is the dropping of the SBS of the orthodontic adhesive despite increase the temperature of the adhesive to 40°C. This could be related to the inflection phenomena that was observed in the dental composite polymers. As it was reported that with increasing dental composite temperatures between (40°C to 45°C), lose their linear correlation length ratio and average order of polymerization. This phenomenon (inflection) represented a glass transition state of the di-methacrylate as a localized vibration and rotational mobility of the bonds and monomer are allowed[5] creating an extensive range of structural conformities that are related to the increased of the thermal motions, creating the detected elevation in the chain correlation length which is represented as an extension, and decrease in their order as polymerization systems conversion is comparatively fast possess a uneven structure[23,29] with an stretched chain conformity, that is simply disturbed and incapable to furtherly extend and re-orientate under thermal disturbance to overcome thermal load. However, the inflection point was reported by Sirovica et al.[30] was between 45- 55°C for the dental composite filling materials.

Also, a significant difference was detected among group 2 and the other tested groups, this could be interpreted by the inflection phenomenon of the dental composite which was noted at certain temperature range as reported by Sirovica et al.[27.] Complete Heating of the adhesive to 40 °C affects the structural conformations of the monomer resulting in significant depression of the SBS.

In orthodontic treatment, the bond between the brackets and tooth surface should withstand the applied mechanical forces and these of mastication and parafunctional movements[31] On the other hand, enamel surface should be render intact during debonding. When the fracture occurs at the enamel- adhesive interface, the score is 0 or 1 while score 2 and 3 denotes that the fracture is at the bracket – adhesive interface[13].

ARI outcomes interpret the SBS results, as during cooling, an increase in the internal cohesion and Van der Waals forces make the adhesive bulkier, resulting to one piece detachment during de bonding, while on heating, the internal cohesion was reduced resulting to fragmental fracture of the adhesive on the brackets and tooth surface during debonding[20 .]/

No significant differences have been recorded when comparing different groups to group 1. Although in some groups the scores of 0 and 1 were more frequent as in group 7. This could be related to rising temperature will reduce adhesive viscosity leading to complete flow of the adhesive into bracket mesh beside improving adhesive radicular mobility for polymerization. As a result, SBS was improved with even detachment of the orthodontic composite after debonding. However, such heating may imply an exceptional bonding of thin less viscose adhesive into the adhesive prime and reduce the internal cohesion and Van der Waals forces but at same extend might have initiated a quick setting process reduced the ARI readings, however; no evidence of enamel stripping was noted[14,20.]

Clinically, the orthodontists who using 3M Transbond Plus™ XT adhesive can store their adhesive in the refrigerator at 5 C˚ precuring to improve the SBS. Composite heater could also be used for improve the SBS when the temperature adjusted at 70 C˚. Additionally, they should avoid to adjusted the temperature at 40 C˚ before polymerization, as this could diminish SBS. Cooling and\ or heating the brackets before curing, could be an additional option to improve the SBS. Besides that, Tooth dryer could be used pre-bonding when an extra option if high SBS is required.

One of the limitations of this study regarding the degree of conversion of the orthodontic composite used in this study, which was not assessed for all groups. Additionally, Intra pulp temperature changes were not measured during using the tooth dryer. Thermo-cycling of the samples after brackets bonding was also not performed. Further study could be conducted to search the effects of these limitations on SBS and ARI.

In conclusion, Storing 3M Transbond Plus™ XT in the refrigerators or cooling the brackets to 5°C before bonding increase the SBS. Additionally, Heating the adhesive and /or bracket base surface pre-curing above 50°C enhance the SBS. Storing the adhesive at 40°C pre-curing should be avoided due to its reverse effects on SBS. Heating the adhesive, brackets and the tooth surface immediately before curing by tooth dryer improves the SBS and reduce ARI.

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Conflict of interest

The authors declare that they have no conflict of interest.

Data availability

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

Author Contribution

Sarmad Sobhi Al-Qassar: concepts, design, definition of intellectual content, literature search, experimental studies, manuscript preparation, editing & review. **Mahmood Kh Ahmed:** design, literature search, clinical studies, experimental studies, data acquisition, manuscript editing & review. **Riyadh Alsaleem:** data analysis, statistical analysis, manuscript editing & review. All the authors actively participated in the manuscript's findings and have revised and approved the final version of the manuscript.

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