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Sara Adel Botros

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EFFECT OF PRE-HEATING TEMPERATURES ON MICROSHEAR BOND STRENGTH AND DEGREE OF CONVERSION OF A DUAL-CURED RESIN CORE FOUNDATION

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ABSTRACT

Aim: To evaluate the effect of pre-heating temperatures of a dual-cured adhesive and a dual-cured resin composite as well as tubule orientation on micro-shear bond strength (μSBS) to dentin. The degree of conversion (DC%) of the adhesive and the composite was also evaluated at the different temperatures.

Materials and Methods: For μSBS, 90 human molars were randomly divided into 18 groups (n=5) according to the three experimental factors. Factor 1: Adhesive temperature, Factor 2: Resin composite temperature; and Factor 3: Tubule orientation (occlusal or axial). Adhesive and composite temperatures were set at 25ºC, 32ºC and 40ºC. The adhesive was applied to dentin according to manufacturer instructions. Each occlusal dentin was stored with four composite micro-cylinders, while each axial dentin was stored with two micro-cylinders. The μSBS testing was run at a crosshead speed of 1mm/min. The DC% of adhesive and composite was evaluated at each temperature using FTIR. Data were analyzed by ANOVA/Tukey's HSD post-hoc test *(P=0.05)*.

Results: Three-Way ANOVA revealed that only "tubule orientation" and "adhesive temperature x composite temperature" had a significant effect on μSBS (*P=0.001* and *P=0.002*, respectively). For occlusal surfaces, "adhesive temperature", "composite temperature" and "adhesive temperature x composite temperature" had no significant effect on μSBS (*P>0.05*). For axial surfaces, "adhesive temperature", "composite temperature" showed no significant effect on μSBS (*P>0.05*), while "adhesive temperature x composite temperature" had a significant effect on μSBS (*P=0.002*). The predominant failure mode was the mixed type (57.43%). The three temperatures tested showed no significant effect on the DC% of the adhesive. On the contrary, pre-heating the resin composite to 40°C revealed a significant higher DC% compared to 25°C.

Conclusion: Tubular orientation seemed to be an influencing factor on μSBS. Raising the temperature to 40°C improved the DC% of dual-cured resin composite, but had no effect on the dual-cured adhesive.

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INTRODUCTION

A growing interest has been recently directed towards simplification of bonding procedures, which led to the introduction of one-step self-etching adhesives,[1] that rely on acidic monomers that cause simultaneous etching and priming by infiltrating the smear layer-covered dentin.[1] A new family of one-step SEAs was launched, the so-called "Universal" or "Multi-mode" adhesives, which could be used in any bonding strategy and are compatible with different substrates.[2]

However, the simplification of such adhesives was achieved at the expense of compromising bond integrity, as their formulations have been rendered more acidic and hydrophilic, hence more permeable to water from the underlying dentin.[3] Chemical incompatibility between the uncured acidic monomers from SEAs and the self-curing components of dual-cured resin composites is also to be considered.[4,5] Hence, manufacturers have developed "dual-cure" adhesive systems, in which co-initiators are incorporated to avoid such adverse reaction.[6]

Resin composites core build-up materials are becoming increasingly popular due to their ability to reinforce remaining tooth structure of severely destructed teeth.[7,8] Dual-cure resin-based composites were marketed in order to overcome the limitations of light attenuation.[9]

Adequate polymerization is an essential factor to obtain proper degree of conversion and mechanical properties of resin-based restorative materials.[10] Previous reports have evaluated the effect of temperature on the polymerization kinetics.[11,12] Raising the temperature of resin composites has been proved to relevantly promote radical activity and monomer mobility, leading to higher polymerization rate.[11,12] Although, manufacturers often recommend refrigeration of SEAs to reduce hydrolytic degradation during storage, low temperature has been shown to increase the viscosity of the adhesive solutions[13] and decrease the evaporation capacity of solvent and water,[14] which would thereby prevent approximation between reactive species.[15] The presence of residual solvent and water in the adhesive layer[16] may also negatively affect bond integrity.[13] Yet to our knowledge, there have been no studies addressing the effect of temperature on the performance of dual-curing self-etch adhesive systems and core build-up resin composites.

Furthermore, it was demonstrated that the direction of dentinal tubules might be an important variable influencing bond strength to dentin,[17] as monomer penetration may be affected by the different tubule orientations.[18,19] In this regard, specimens will be prepared with the bonding surface either perpendicular or parallel to the dentinal tubules in order to gain a better understanding of bonding mechanisms in relation to dentin location.

The aim of this study was to evaluate: 1) The effect of dual-cured adhesive and dual-cured resin composite temperatures as well as tubule orientation on μSBS to dentin; 2) the effect of dual-cured adhesive and dual-cured resin composite temperatures on their DC%. The null hypothesis tested were: 1) Adhesive temperature and resin composite temperature as well as Tubule orientation had no effect on μSBS to dentin. 2- Neither adhesive temperature nor resin composite temperature had an effect on their DC%.

MATERIALS AND METHODS

One dual-cured universal adhesive (Futurabond U/FBU) and one dual-cured core build-up resin composite (Rebilda DC/RDC) were used in this study. Material/manufacturer, description, composition, and lot number are presented in Table 1.

Micro-Shear Bond Strength (μSBS)

Experimental grouping

A total of 90 non-carious freshly extracted human molars were collected. They were assigned to 18

TABLE (1): Material/Manufacturer, Description, Composition and Lot number.

Material/ Manufacturer	Description	Composition	Lot number
Futurabond U (FBU), VOCO GmbH, Cuxhaven, Germany	-Dual-cured universal adhesive -pH≈2.3	**-Liquid 1:** HEMA, Bis-GMA, HEDMA, UDMA, fumed silica, acid modified methacrylates, camphorquinone, BHT, amine. **-Liquid 2:** ethanol, water, initiator, DC catalyst.	#1801167
Rebilda DC (RDC), VOCO GmbH, Cuxhaven, Germany	-Dual-cured core build-up resin composite -Dentin shade	UDMA, DDMA, Bis-GMA, HEDMA, BHT, dibenzoyl peroxide, camphorquinone, silica, bariumborosilicate glass ceramic, accelerators.	#1750122
HEMA: ***2-Hydroxyethyl methacrylate,*** **Bis-GMA:** ***Bisphenol A diglycidyl methacrylate,*** **HEDMA:** ***Hydroxyethyl dimethacrylate,*** **UDMA:** ***Urethane dimethacrylate,*** **BHT:** ***Butylhydroxytoluene,*** **DDMA:** ***Dodecyldimethylamine.***			

experimental groups (n=5 teeth/group) according to the three experimental factors of the study: 1) *Adhesive temperature:* 25ºC (room temperature),[20] 32ºC (tooth simulated temperature)[21] or 40ºC (pre-heating temperature).[22] 2) *Composite temperature:* 25ºC (room temperature),[23] 32ºC (tooth simulated temperature) or (pre-heating temperature).[24] 3) *Tubule orientation:* occlusal and axial surfaces.

Teeth preparation

Soft and hard deposits were removed using ultrasonic scaler (P5 Booster, Acteon Satelec, Italy). For occlusal bonding, the occlusal surfaces of 45 teeth were horizontally cut exposing superficial dentin using low speed diamond discs with copious water irrigation. While for axial bonding, the crowns of 45 teeth were vertically sectioned mesiodistally, to get 90 buccal and lingual halves. The roots were cut off at the level of cementoenamel junction.

The dentin of both occlusal and axial surfaces were ground using a high speed yellow-coded diamond bur (TR-25EF, Mani Dia-Burs, Japan) under air/water spray to create a standardized smear layer.

Bonding procedures

Single doses of FBU were heated to the desired temperature in an incubator (BT 1010, Biotech Company, Egypt) for 10 minutes. The single dose unit was activated as described by the manufacturer. The adhesive was stirred with the micro-brush applicator for 2 seconds, to create a homogenous mix (Futurabond U, Instructions of use, VOCO). It was then applied to the dentin surface, rubbed for 20 seconds and gently air-dried with oil-free compressed air for 5 seconds according to manufacturer's instructions. Prior to light-curing of the adhesive, 4 stainless steel metal tubes (1.2mm internal diameter x 1 mm height) were placed at least 2 mm apart over the occlusal dentin surfaces. While only 2 metal tubes were placed over each half of axial dentin surfaces, at least 2 mm away from pulp chamber. The tip of a LED light-curing device (Elipar, 3M ESPE, USA), with an output of 1200 mW /cm^2, was placed over the tubes and the adhesive was light-cured for 10 seconds.

Rebilda DC composite cartilage was heated to the desired temperature using another incubator and left for 10 minutes. Resin composite was injected into each metal tube using the automix tip supplied by the manufacturer. A sharp explorer was used to extrude the voids within the resin composite inside each tube. The light-curing device was placed as close as possible to the tubes and activated initially for 20 seconds, then through a Mylar strip (TOR VM, Russia) for another 20 seconds. The specimens

were allowed to set for 5 minutes in 100% relative humidity before storage. The specimens were stored in distilled water at room temperature for 48 hours before μSBS testing.

Micro-shear bond strength testing

Each specimen was fixed over a rectangular acrylic block (Acrostone, Egypt) using cyanoacrylate glue, which was attached to the lower jig of a universal testing machine (INSTRON 2519-104, Illinois, USA). A 0.5 mm stainless steel orthodontic wire (TRU-CHROME, RMO, USA) was wrapped semi-circularly around the bonded assembly as close as possible to composite/dentin interface and aligned with the loading axis of the upper movable jig of the testing machine. Micro-shear test was run at a crosshead speed of 1 mm/min until failure. The shear bond strength was calculated by dividing the load (Newtons) over the respective surface area (mm^2).

Each de-bonded dentin surface was evaluated for fracture analysis using a light microscope (ZEISS Primo Star, Germany). The fracture modes were classified as follow:

1. Adhesive failure: The fracture occurred at the adhesive/dentin interface.
2. Mixed failure: The fracture occurred at the adhesive/dentin interface accompanied with part of the resin composite left on the dentin surface.
3. Cohesive failure: The failure occurred within the resin composite or dentin.

Degree of Conversion (DC%)

Degree of conversion of Futurabond U

A total of 15 Potassium Bromide KBr pellets were divided into 3 groups (n=5) according to the three adhesive temperatures previously mentioned in μSBS test. At each respective temperature, FBU was uniformly applied to KBr pellet surface using the micro-brush applicator and rubbed for 20 seconds. The adhesive layer was air-dried for 5 seconds and light-cured for 10 seconds with the LED light-curing device. All pellets were stored dry in light-proof containers for 48 hours before the evaluation of the DC%.

Fourier Transform Infrared Spectrometer (FT-IR) spectra (JASCO 6800, Japan) of the unpolymerized adhesive were recorded in the absorbance mode. For calculating DC%, the percentage of unreacted C=C double bonds was determined from the ratio of absorbance peak areas of aliphatic C=C double bonds (peak at 1638 cm^{-1}) against aromatic component (peak at 1608 cm^{-1}) which was used as an internal standard before and after curing. The underlying peak area was calculated for each peak, using a standard baseline technique with the aid of computer software program provided with the spectrometer (JASCO Spectral Analysis).

The degree of monomer conversion was determined using the following equation:

$$DC\% = 1 - \left(\frac{[\text{abs (aliphatic C=C) /abs (aromatic C=C)}]\ polymer}{[\text{abs (aliphatic C=C)/abs (aromatic C=C)}]\ monomer} \right) \times 100$$

Degree of conversion of Rebilda DC

A total of 15 resin composite cylinders were fabricated and assigned to 3 groups (n=5) according to the three resin composite temperatures. A double-faced adhesive tape with a 2 mm central hole was placed over a polyacetate sheet. A clear polyethylene tube (2 mm internal diameter x 2 mm height) was fixed over the sheet. At each respective temperature, RDC was injected into the tube through the automix tip and an explorer was used to eliminate any air bubbles entrapped. A clear a polyacetate sheet was pressed gently over the tube to remove excess material. It was light-cured for 40 seconds with the

LED light-curing device. The tubes were stored in light-proof containers for 48 hours. The tube was cut off using a Bard Parker blade #15 and the top surface was marked with an indelible pen at its side.

The uncured resin composite mix was smeared on a KBr pellet to record a spectrum of uncured material. Each polymerized cylinder was scraped with a scalpel into fine powder, mixed with KBr powder in 1:10 ratio and the spectra was recorded in the absorbance mode. DC% was calculated following the same method previously mentioned for FBU.

Statistical Analysis

Statistical analysis was performed using IBM SPSS Statistics Version 2.0 for Windows. Data was presented as mean and standard deviation (SD). The significance level was set at P=0.05. Kolmogorov-Smirnov and Shapiro-Wilk tests were used to assess data normality.

For μSBS, Three-Way ANOVA was used to evaluate the effect of the three investigated variables and their interactions. Two-Way ANOVA was performed to evaluate the effect of adhesive and composite temperatures and their interaction on μSBS of occlusal and axial groups. Intergroup comparisons between μSBS of occlusal and axial groups were conducted using Independent Student-t test. Intragroup comparisons within each adhesive and composite temperature group, regarding μSBS and DC%, were done using One-Way ANOVA followed by Tukey's HSD post-hoc test.

RESULTS

Microchear Bond Strength

Three-Way ANOVA (Table 2) showed that neither the adhesive nor the composite temperature had a significant effect on μSBS (*P=0.290* and *P=0.133*, respectively). However, the tubule orientation (*P=0.002*) showed a significant effect on μSBS. Only the interaction between "adhesive temperature x composite temperature" had a significant effect on μSBS (*P=0.001*).

For occlusal bonding, Two-Way ANOVA (Table 3) showed that "adhesive temperature", "composite temperature" and their interaction had no statistically significant effect on μSBS (*P=0.876*, *P=0.499* and *P=0*.087, respectively).

TABLE (2): Three-Way ANOVA for the effect of adhesive temperature, composite temperature, tubule orientation and their interaction on μSBS to dentin.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Adhesive temperature	29.232	2	14.616	1.257	0.290
Composite temperature	48.219	1	24.109	2.074	0.133
Tubule orientation	119.350	2	119.350	10.265	0.002
Adhesive temperature x Composite temperature	231.288	4	57.822	4.973	0.001
Adhesive temperature x Tubule orientation	6.424	2	3.212	0.276	0.759
Composite temperature x Tubule orientation	11.703	2	5.851	0.503	0.607
Adhesive temperature x Composite temperature x Tubule orientation	52.478	4	13.120	1.128	0.350

TABLE (3): Two-Way ANOVA for the effect of adhesive temperature, composite temperature and their interaction on μSBS to occlusal surfaces.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Adhesive temperature	35.112	2	17.556	1.383	0.876
Composite temperature	51.324	2	25.662	2.021	0.499
Adhesive temperature x Composite temperature	235.512	4	58.878	4.637	0.087

TABLE (4): Mean ± Standard Deviation for the effect of different adhesive and composite temperatures on μSBS (MPa) of occlusal surfaces.

		Adhesive temperature		
		25°C	**32°C**	**40°C**
Composite temperature	**25°C**	17.25 ± 4.0^{aA}	18.02 ± 2.88^{aA}	22.04 ± 4.86^{aA}
	32°C	21.04 ± 4.07^{aA}	20.06 ± 5.82^{aA}	17.98 ± 0.96^{abA}
	40°C	18.69 ± 4.92^{aA}	19.79 ± 3.36^{aA}	15.66 ± 3.28^{bA}

Means with same superscript small letters within each column and capital letters within each row are not statistically significantly different at $P \leq 0.05$.

One-Way ANOVA and Tukey's HSD post-hoc test (Table 4) showed that within 25°C and 32°C adhesives, there was no statistically significant difference between the different composite temperatures (*P>0.05*). While, for 40°C adhesive, there was a statistically significant difference between 25°C and 40°C composites (*P<0.05*). Within each composite temperature, there was no statistically significant difference between the three tested adhesive temperatures (*P>0.05*).

For axial bonding, Two-Way ANOVA (Table 5) showed that "adhesive temperature" and "composite temperature" had no significant effect on μSBS (*P=0.125* and *P=0.081*, respectively). While their interaction showed a significant effect on μSBS (*P=0.002*).

One-Way ANOVA and Tukey's HSD post-hoc test (Table 6) showed that within 25°C adhesive, there was no statistically significant difference between the different composite temperatures (*P>0.05*). Within 32°C adhesive, 32°C composite showed the significantly higher μSBS mean values compared to 25°C and 40°C (*P<0.05*), which were statistically similar (*P>0.05*). Within 40°C adhesive, 25°C composite showed significantly higher μSBS mean values compared to 32°C (*P<0.05*), while the 40°C composite had no statistically significant difference from 25°C and 32°C (*P>0.05*).

Within 25°C and 40°C composites, there was no statistically significant difference between the different adhesive temperatures (*P>0.05*). Whereas, within 32°C composite, there was no statistically significant difference between 25°C and 32°C adhesives (*P>0.05*), which had significantly higher μSBS mean values compared to 40°C adhesive (*P<0.05*).

Independent Student-t test (Table 7) showed that there was only a statistically significant

difference between occlusal and axial dentin surfaces at 40°C adhesive temperature x 40°C composite temperature, where the axial surface yielded the highest μSBS values (*P=0.028*).

Fractographic analysis

The overall fractographic analysis showed that the adhesive type represented 31.49%, the mixed type was 57.43%, the cohesive failure in composite was 6.71% and the cohesive failure in dentin was 4.37%.

The failure analysis of occlusal surfaces (Figure 1) revealed that the predominant failure mode was the mixed type (65.70%); followed by adhesive type (23.83%), cohesive failure in composite (9.35%) and cohesive failure in dentin (1.16%).

While for the axial surfaces (Figure 2), the predominant failure mode was the mixed type (49.10%); followed by adhesive type (39.18%), cohesive failure in dentin (7.60%) and cohesive failure in composite (4.07%).

Degree of Conversion

One-Way ANOVA and Tukey's HSD post hoc test (Table 8) showed that for FBU, there was no significant difference in DC% between the different tested temperatures (*P>0.05*). For RDC, showed that DC% at 40°C was statistically significantly higher than at 25°C (*P<0.05*). While, there was no significant difference in DC% between 25°C and 32°C and between 32°C and 40°C (*P>0.05*).

TABLE (5): Two-Way ANOVA for the effect of adhesive temperature, composite temperature and their interaction on μSBS of axial surfaces.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Adhesive temperature	8.863	2	4.432	0.299	0.125
Composite temperature	30.812	2	15.406	1.038	0.081
Adhesive temperature x Composite temperature	117.779	4	29.445	1.983	0.002

TABLE (6): Mean ± Standard Deviation for the effect of different adhesive and composite temperatures on μSBS (MPa) to axial surfaces.

		Adhesive temperature		
		25°C	**32°C**	**40°C**
Composite temperature	**25°C**	20.06 ± 2.32^{aA}	19.016 ± 3.70^{bA}	22.02 ± 2.28^{aA}
	32°C	23.75 ± 2.70^{aA}	25.49 ± 3.72^{aA}	18.21 ± 1.39^{bB}
	40°C	20.28 ± 2.22^{aA}	21.96 ± 1.87^{bA}	20.21 ± 2.58^{abA}

Means with same superscript small letters within each column and capital letters within each row are not statistically significantly different at P≤ 0.05.

TABLE (7): Intergroup comparisons of μSBS mean values (MPa) between occlusal and axial surfaces within each adhesive and composite temperature interaction.

Adhesive temperature x Composite temperature	Occlusal	Axial	P-value
25°Cx25°C	17.25 ± 4.0	20.06 ± 2.32	0.212
32°Cx25°C	18.02 ± 2.88	19.01 ± 3.70	0.649
40°Cx25°C	22.04 ± 4.86	22.02 ± 2.28	0.996
25°Cx32°C	21.04 ± 4.07	23.75 ± 2.70	0.236
32°Cx32°C	20.06 ± 5.82	25.49 ± 3.72	0.117
40°Cx32°C	17.98 ± 0.96	18.21 ± 1.39	0.770
25°Cx40°C	18.69 ± 4.92	20.28 ± 2.22	0.531
32°Cx40°C	19.79 ± 3.36	21.96 ± 1.87	0.242
40°Cx40°C	15.66 ± 3.28	20.21 ± 2.58	0.028

P-value: independent t-test between occlusal and axial surfaces within each adhesive and composite temperatures at P≤ 0.05.

TABLE (8): Mean ± Standard for the effect of different temperatures on DC% of Futurabond U and Rebilda DC.

Temperature	Futurabond U	Rebilda DC
25°C	67.47 ± 2.13^{a}	62.52 ± 3.12^{B}
32°C	67.52 ± 4.19^{a}	64.81 ± 2.15^{AB}
40°C	68.85 ± 6.21^{a}	68.61 ± 2.91^{A}

Means with same superscript small letters within Futurabond U and capital letters within Rebilda DC are not statistically significantly different at P≤ 0.05.

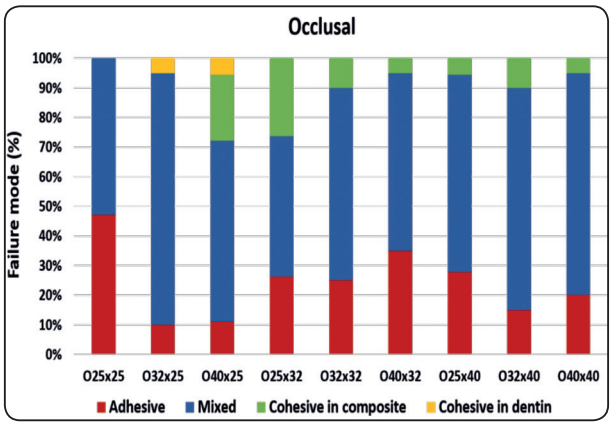

Fig. (1) Stacked column bar showing the percentage distribution (%) of each failure mode in occlusal bonding groups.

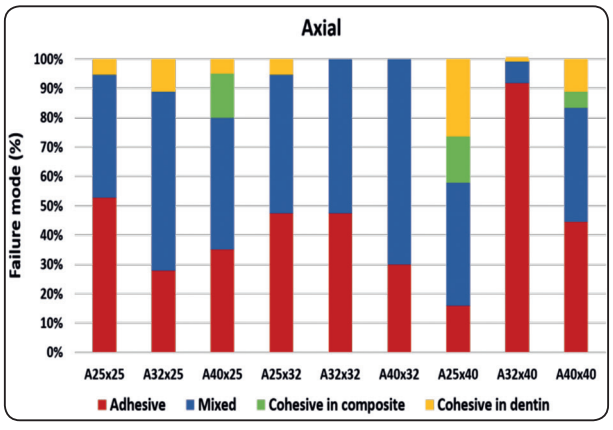

Fig. (2): Stacked column bar showing the percentage distribution (%) of each failure mode in axial bonding groups.

DISCUSSION

A new group of dental adhesives has been introduced to the market as ''universal'' adhesives, which were originally one-step SE adhesives.[2] Universal adhesives contain specific carboxylate or phosphate monomers that bond chemically to dental substrates.[25]

Adequate polymerization of dental adhesives and resin composites has been considered an essential requisite for good clinical performance. However, in light-cured adhesives, achieving proper polymerization might be difficult for regions remote from the light-curing source,[26] Considering the light attenuation factor, dual-curing adhesive systems and core build-up composite materials have been launched in the market to enhance the efficiency of polymerization reaction.[6,9]

Any attempts to improve the degree of conversion of resin-based restorative materials would positively contribute to their immediate bonding performance and long-term durability.[27,28] Pre-heating of adhesive systems and resin composites has been reported in several studies to be a good way of achieving a better DC% by promoting radical activity and accelerating monomer mobility, leading to higher polymerization rate.[27,29]

Most in-vitro tests evaluating interfacial bond strength of adhesive dental materials were generally conducted at room temperature conditions (approximately 25ºC).[20,23,30] These conditions are far from what occurs in the oral cavity, as the average oral temperature was found to be 30°C[31] and the temperature of a prepared tooth ranges from 27.8ºC to 32ºC.[21] For that matter, it was important to evaluate the effect of representative temperatures of different clinical conditions (25ºC: room temperature, 32ºC: simulated prepared tooth temperature, and 40ºC: pre-heating temperature) on dentin μSBS and DC% of a commercial dual-cure resin core foundation system. To date, there have been no published studies investigating the impact of temperature on the performance of dual-cure adhesives and core build-up resin composites.

Dentinal tubules are arranged in different directions depending on their location in the tooth.[18,32] The effect of these regional variations on dentin biomechanical function are of importance to dentin bonding. A 20% variation in the tensile strength of dentin has been observed, depending on whether the tubules were aligned perpendicular or parallel to the force axis.[33]

Micro-shear bond strength testing remains a useful method for brittle substrates, since the specimens are not pre-stressed prior to testing.[34] It also allows for regional mapping and depth profiling of different substrates.[34] The orthodontic-wire loop method has showed less variability in microshear results and was easier to use than the chisel method.[35]

Regarding μSBS results, the null hypothesis was partially rejected, as only the tubule orientation had shown to have a significant effect on μSBS. Several studies[36-38] have reported that the bond strength of the bonded interface parallel to the direction of the tubules was higher than that with tubules cut perpendicularly. This could be in disagreement with the findings of the present study. There were no significant differences observed between occlusal and axial surfaces except for 40°Cx40°C groups, though the numerical μSBS mean values tended to be slightly higher in the axial groups. It was depicted that the arrangement of the collagen fibrils, which run perpendicularly in relation to the dentinal tubules, along with the parallel arrangement of apatite crystals with the long axis of collagen fibrils, might be the possible reason of such higher bond strength in axial wall bonding.[39] This structural organization would render the dentin substrate 1.43 times more resistant to failure parallel to dentinal tubules than perpendicular to them.[40] These findings could further explain the increased percentage of cohesive failure in dentin in the axial groups.

A study[41] has found that when the pH of an experimental self-etching adhesive was higher than 2.3, the bond strength had no difference between the occlusal and axial positions. The results of the

previous study were in agreement with the findings of this study. Futurabond U employed in the current research is a universal adhesive with a pH of ≈2.3, which is categorized as a mild self-etching adhesive.[1]

Concerning μSBS results of occlusal dentin surfaces, there were no significant differences within the composite temperature groups. In Finite Element Analysis, it was concluded that the stresses distributed in μSBS test at the interface angle between resin composite and dentin.[42] Placido et al.,[42] reported that the load applied by a wire-loop might develop a bending moment during testing. Hence, it could be hypothesized that bending of the specimen could depend on the length of the resin composite cylinder as well as on its mechanical properties. Since the resin composite cylinders prepared in this study were 1 mm in thickness, the bending moment might not have a valuable impact on their bond strength results, resulting in insignificant bond strength values between the different resin composite temperatures tested.

The results of this study revealed that when the adhesive temperature was raised up to 40°C, the bond strength dropped especially when the resin composite temperature was 40°C. Studies have indicated that the oxygen-inhibited layer (OIL) of the cured adhesive increased the bond strength by co-polymerization with the overlying resin composite to produce a chemical bond.[43] At room temperature, the viscous nature of OIL, owing to the presence of unreacted methacrylate groups,[44] might make FBU readily adapt and blend with the overlying RDC, regardless of its temperature. The free radicals in RDC could subsequently create an interpenetrating network, or in other words, an interdiffusion zone with the uncured monomer mixture within OIL.[43,44] On the contrary, warming FBU to 40°C along with the energy and heat generated from the light curing device, might have driven the polymerization reaction of OIL by activating the unreacted photoinitiators and thereby consuming unreacted monomers to some extent.[44]

With reference to μSBS results of axial dentin surfaces, when RDC was warmed to 32°C and FBU to 40°C, the bond strength had significantly declined compared to 25°C and 32°C adhesive temperatures and 25°C resin composite temperature. Consumption of unreacted monomers in OIL, even to a limited extent,[44] could increase by heating the adhesive to 40°C. By warming RDC up to 32°C, the benzoyl peroxide might start to prematurely react with tertiary amines during the time of dispensing,[45] which was responsible for the observed increase in viscosity of the extruded material. This might have interfered with proper flow of the heated composite within the narrow metal tubes; thereby worsening its adaptation to dentin surface. Moreover, this premature setting reaction of RDC could have presumably consumed the available radicals prior to light activation; which resulted in a limited reaction with OIL.

Regarding DC%, the null hypothesis should be partially rejected, as the pre-heating temperature revealed a significant effect only on the DC% of dual-cured resin composite. For FBU, the DC% results showed no significant differences at the three tested temperatures. When FBU was warmed to the respective temperatures, solvent might get evaporated as soon as the adhesive solution was mixed with the micro-brush, resulting in proper solvent/water removal. Moreover, the continuous rubbing of the adhesive over the KBr pellets for 20 seconds, could have enhanced further evaporation of the solvent/water mixture. This could clarify why the DC% of FBU was not affected by the temperature elevation.

It was formerly demonstrated that the beneficial effect provided by pre-heating the resin composite in terms of improved DC% was much less pronounced after 24 hours, when compared with immediate DC% evaluation.[46] It was also argued that when a higher conversion rate was reached immediately, further enhancement in DC% was improbable due to the restriction of monomer mobility.[46] The results of the present study were in disagreement with these above-mentioned reports. These studies have

evaluated light-cured resin composites; whereas, a dual-cure resin composite was used in this study. Warming the DC resin composite to 40°C might hastened the chemical curing mechanism during mixing the resin composite within the automix tip. The enhancement of the chemical reaction could thereby induce an improvement in the DC% of the DC resin composite. This could explain the increase in DC% of Rebilda DC at 40°C compared to 25°C.

It should be highlighted that in this study, the pre-heating process of the adhesive and resin composite was performed under a controlled condition. However, all other steps of bonding procedures and DC% evaluation were performed at room temperature, which presented a drawback of this study as they were not accomplished under strict controlled conditions. This could have affected the results of this study in some way. It should be advised that in future research, all preparation steps should be performed at a controlled temperature similar to the pre-heating target temperature. This should be including the application technique, adhesive air-drying, light-curing of adhesive and resin composite and, if possible, the temperature of the tooth used itself.

CONCLUSIONS

Under the limitations of this study, several conclusions could be suggested:

1. The orientation of dentinal tubules showed to be an influencing factor on μSBS of dual-cured universal adhesive/dual-cured resin composite to dentin.
2. Warming up the dual-cured adhesive to 40°C negatively affected the μSBS of 40°C dual-cured resin composite to occlusal dentin. On the other hand, heating resin composite to 32°C significantly reduced the μSBS to axial dentin, when the adhesive was warmed to 40°C.
3. Raising the temperature up to 40°C positively influenced the DC% of dual-cured resin composite, but had no effect on the dual-cured adhesive.

Clinical significance: Pre-heating of dual-cured adhesive or dual-cured resin composite seemed to be an unnecessary step to improve bond strength to dentin; but raising the dual-cured resin composite temperature to 40°C showed to be crucial to improve its DC%.

Conflict of interest: The authors had no conflict of interest in the materials used in this study.

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