Original Article

Effect of preheating nano-hybrid and bulk-fill composites with warm airstream on their microtensile bond strength to dentin

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Abstract

Aims: This study assessed the effect of preheating nano-hybrid and bulk-fill composites with warm airstream on their microtensile bond strength (MTBS) to dentin.

Materials and Methods: In this *in vitro* study, dentin was exposed in 42 extracted premolars that were assigned to two groups ($n = 21$) for bonding to EverX Posterior bulk-fill and Grandio nano-hybrid posterior composite. Each group was subdivided into three subgroups for preheating of composite to 50°C by a commercial composite warmer (Subgroup 1), a warm airstream by a hair dryer for 10 s (Subgroup 2), and no heating (room temperature; Subgroup 3). Composite cylinders with a 4-mm height were bonded to the tooth surface with a 5th-generation bonding agent using the incremental technique and cured (each increment for 30 s). The MTBS to dentin was measured, and the bonding interface was evaluated under a stereomicroscope and a scanning electron microscope (SEM). Data were analyzed using one-way and two-way ANOVA, Tukey's test, and independent *t*-test (α =0.05).

Results: In both composite groups, the mean MTBS of the warm airstream subgroup was significantly higher than that of warmer ($P < 0.001$) and control ($P < 0.001$) subgroups. The mean MTBS of the warmer subgroup was significantly lower than that of control subgroup ($P = 0.01$). The MTBS of the EverX composite was significantly higher than that of the Grandio composite only in the warmer group ($P < 0.05$). Adhesive failure occurred more frequently in all groups with the highest frequency in airstream subgroup of both composite types.

Conclusion: Preheating with a warm airstream significantly increased the MTBS of both composite types to dentin.

Keywords: Composite resins; EverX Posterior; Grandio; preheating

INTRODUCTION

Since composite resins' introduction in 1950, efforts have focused on enhancing their mechanical properties.[1] However, issues such as significant polymerization shrinkage lead to concerns such as marginal leakage, increased posttreatment pain, and high water sorption.[2] Cracks, fractures, and secondary caries often necessitate the replacement of these restorations.[3] The durability of composite restorations hinges on their bond strength to tooth structures,[4] with a strong bond ensuring improved retention, reduced microleakage, and extended clinical longevity.

Increasing the degree of conversion (DC) without adversely affecting the marginal adaptation is highly important to

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optimize the properties of composite resins. Besides other strategies to address this, such as soft-start lighting, the preheating of the composite serves as another solution.[5] It has been reported that preheating the composite to 60°C can increase the DC of a composite at the surface and 2 mm subsurface.[6] Increasing the composite temperature under isothermal conditions can increase the cross-linking of monomers and their reactivity, and thus enhance the DC. A heightened DC not only strengthens mechanical properties but also reduces solvent sorption and degradation in the oral setting.[4] Furthermore, research indicates that warming the composite to 60° results in a pulp temperature elevation of <1°. Notably, this increase remains below the critical threshold for pulp temperature, even lower than the rise induced by light curing.[7]

The majority of available studies on composite preheating have an *in vitro* design and utilize a warmer for preheating the composite resins. However, the time lapse between the removal of the composite from the warmer and its application in the cavity can affect the results. It has been reported that the temperature of a 60° composite specimen can drop by 50% within 2 min following removal from the warmer.[4] To counteract this temperature loss, this study evaluated an alternative strategy, using a warm airstream from a modified hair dryer after applying the resin material into the cavity, to keep the composite warmth during application until light-curing in order to increase restoration longevity. Considering all the above, this study aimed to assess the effect of preheating a nano-hybrid and a bulk-fill composite with a warm airstream on their microtensile bond strength (MTBS) to dentin.

MATERIALS AND METHODS

This *in vitro* experimental study was conducted on 42 human premolars extracted for purposes unrelated to this study, such as orthodontic treatment. The study protocol was approved by the ethics committee of the university (IR.MUMS.DENTISTRY.REC.1401.090).

Sample size

The sample size was calculated to be 7 in each subgroup (a total of 42) according to a previous study[8] assuming $\alpha = 0.05$, $\beta = 0.08$, and effect size of 0.25.

Eligibility criteria

Extracted human premolars with no caries, fractures, or dental anomalies were included in the study.

Intervention

The teeth were immersed in distilled water containing 0.5% chloramine T solution for 1 week. They were then sectioned below the dentinoenamel junction by an automatic feed bench lathe machine to expose the dentin. Next, the teeth were randomly assigned to two groups ($n = 21$) based on the type of composite to be applied [Table 1]:

- Group 1: Bonding to EverX Posterior bulk-fill composite (GC, Tokyo, Japan)
- Group 2: Bonding to Grandio nano-hybrid posterior composite (VOCO, Cuxhaven, Germany).

Each group was then subdivided into three subgroups based on the temperature of the composite before curing:

- Subgroup 1: Preheating of composite to 50°C in a commercial composite warmer (Smileline, Tallahassee, Florida, USA)
- Subgroup 2: Preheating of composite with warm airstream by using a modified hair dryer (Maxi, Italy)

Figure 1: Checking the output temperature of the modified hair dryer with a thermometer

Table 1: Characteristics of composite resins used in this study

Composite resin	Type	Manufacturer	Composition	Filler percentage by weight and volume	Color	Lot number
Grandio	Nano-hybrid	VOCO, GmbH, Cuxhaven	Resin Matrix: Bis-GMA, dimethacrylate, UDMA, TEG-DMA Filler: Silicium dioxide nanofillers (20–50 nm) glass-ceramic microfillers (1 μm)	87 wt% 71.4 v%	A2	1918383
EverX posterior	Bulk-fill fiber-reinforced composite	GC Europen, Leuven, Belgium	Resin matrix: SemiIPN: netpoly (methyl methacrylate)-inter-net-poly (bis-glycidyl-A-dimethacrylate): Bis-GMA, TEGDMA, and PMMA Fillers: E-glass fiber, barium borosilicate Bis-GMA, PMMA, TEGDMA	74.2 wt% 53.6 v%	Universal	2202141

UDMA: Urethane dimethacrylate, TEG-DMA: Triethylene glycol dimethacrylate, SemiIPN: Semi-interpenetrating polymer network, PMMA: Polymethyl methacrylate, Bis-GMA: Bisphenol A-glycidyl methacrylate

with adjustable temperature and a modified head for 10 s. The modified hair dryer was equipped with a temperature monitor. Following modifications to the tip, the output temperature was assessed using a thermometer to confirm stability [Figure 1]
- Subgroup 3: No preheating (room temperature composite).

In all groups, composite resins were bonded to the teeth using Single Bond 2 (3M, USA) a 5th-generation bonding agent. For this purpose, acid etching was performed for 15 s as instructed by the manufacturer, and after rinsing with air and water spray, the teeth were dried using air spray for 15 s. Two layers of bonding agent were applied for 10 s. Solvent evaporation was performed from 20 cm distance for 10 s. Light curing was performed with a light emitting diode (LED) curing unit (LED D; Woodpecker, Zhengzhou Linker Medical Equipment Co., China) with 600 mW/cm^2 energy density for 20 s. Composite cylinders were bonded to the teeth using a 4 mm mold. The composite resin was applied into the mold in 1 mm increments. Each increment was independently cured for 30 s. All restorations were carried out by one clinician at 25°C. The restored teeth were stored in distilled water at 37°C for 24 h, after which they were mounted and sectioned for the MTBS test. The MTBS of each section was measured using a universal testing machine (ZWICK/ROELL ZO20, Germany) at a crosshead speed of 1 mm/min.

Mode of failure

Stereomicroscopic assessment

The teeth were longitudinally split in half and placed in an ultrasonic bath containing EDTA for 380 s. They were then inspected under a stereomicroscope (Dino-lite Pro, AnMo Electronics Corp, Taiwan) at ×200 magnification.

Scanning electron microscopic (SEM) assessment

After sandblasting, one specimen from each group was gold sputter-coated for Scanning electron microscope (SEM) assessment (FEI XL30 SEM, USA) with 10 kV voltage at ×2000 magnification.

Statistical analysis

The Shapiro–Wilk test was applied to analyze the normality of data distribution, which showed normal distribution of data. Thus, two-way ANOVA was used to analyze the effects of preheating and composite type on MTBS of composite to dentin. Considering their significant interaction effect ($P = 0.001$), the effect of preheating on MTBS was analyzed by one-way ANOVA followed by the Tukey's test. An independent t-test was employed to analyze the effect of composite type on MTBS. The Fisher's exact test was used to compare the frequency of different modes of failure. The level of statistical significance was set at 0.05.

RESULTS

Microtensile bond strength

Table 2 presents the measures of central dispersion for the MTBS of the two composites in the three groups. Two-way ANOVA showed a significant interaction effect of preheating and composite type on MTBS of composite to dentin ($P = 0.001$). Thus, the effect of preheating on MTBS was analyzed by one-way ANOVA, which showed that preheating of both composite types significantly impacted their MTBS to dentin ($P < 0.001$ for both).

Pairwise comparisons of the subgroups [Table 3] showed that in both composite types, the MTBS in warm airstream group was significantly higher than that in warmer and control subgroups ($P < 0.05$), and the MTBS of the warmer subgroup was significantly lower than that of control subgroup ($P < 0.05$). An independent t-test was employed to analyze the effect of composite type on MTBS, which showed that only in a warmer group, the MTBS of EverX composite was significantly higher than that of Grandio composite ($P < 0.05$); however, the difference between the two composite types in MTBS was not significant in warm airstream and control groups ($P > 0.05$).

Failure mode

Table 4 presents the frequency of different failure modes in the study groups. The frequency of cohesive failure was absent in all groups. Adhesive failure had a higher frequency in all groups with the highest frequency in airstream subgroup of both composite types. The Fisher's exact test showed a significant difference in the frequency of different failure modes among the groups ($P = 0.032$).

Stereomicroscopic and SEM assessments

Stereomicroscopic assessments at ×200 magnification [Figure 2] and SEM assessments at ×2000 magnification [Figure 3] revealed no significant difference among the

Table 2: Measures of central dispersion for the microtensile bond strength (MPa) of the two composites in the three groups

Group	*n*	Grandio			EverX			*P**
		Mean±SD of MTBS	Minimum	Maximum	Mean±SD of MTBS	Minimum	Maximum	
Control	18	7.42±2.92	3.17	13.7	9.58±3.58	4.93	18.68	0.056
Warmer	18	4.48±1.04	3.30	6.53	5.70±1.23	3.39	7.91	0.003
Airstream	18	17.68±4.09	12.48	27.62	19.50±4.07	13.01	28.62	0.189
*P***		<0.001			<0.001			

*Independent *t*-test, **One-way ANOVA. SD: Standard deviation, MTBS: Microtensile bond strength groups; however, the interface was noticeably more uniform and had a superior adaptation in the warm airstream subgroups. The control and warmer subgroups exhibited higher porosities and occasional cracks at the bonding interface or within the composite and dentin.

Table 3: Pairwise comparisons of microtensile bond strength of the subgroups in each composite group by the Tukey's *post hoc* test

Composite	Subgroup	SE	Mean difference	*P*
Grandio	Airstream-warmer	0.99	13.20	<0.001
	Airstream-control	0.99	10.25	<0.001
	Warmer-control	0.99	2.94	0.012
EverX	Airstream-warmer	1.07	13.8	<0.001
	Airstream-control	1.07	9.92	<0.001
	Warmer-control	1.07	3.88	0.002

SE: Standard error

Table 4: Frequency of different failure modes in the study groups

Group	Adhesive, *n* (%)	Cohesive	Mixed, *n* (%)	Fisher's exact test
Grandio				
Control	16 (88.9)	0	2 (11.1)	*P*=0.032
Warmer	14 (77.8)	0	4 (22.2)	
Airstream	17 (94.5)	0	1 (5.5)	
EverX				
Control	17 (94.5)	0	1 (5.5)	
Warmer	16 (88.9)	0	2 (11.1)	
Airstream	18 (100)	0	0	

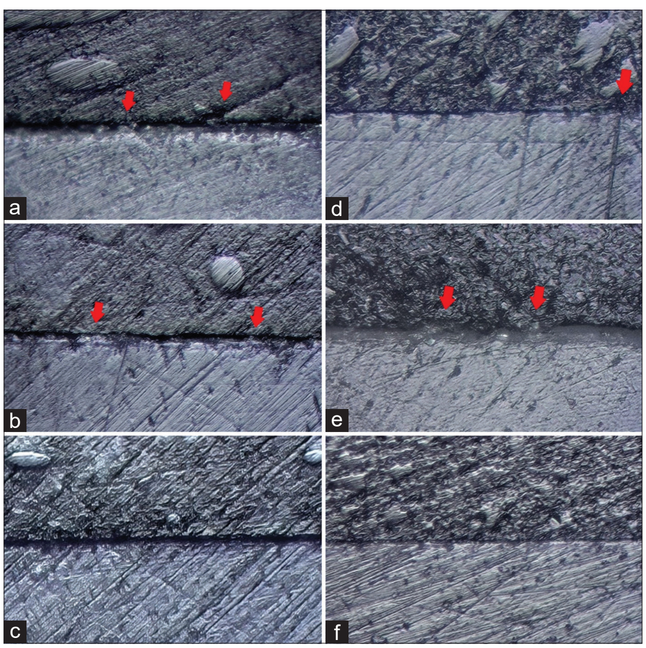

Figure 2: Stereomicroscopic assessment at ×200 magnification; (a) interface of EverX control subgroup; red arrows point to defects; (b) interface of EverX warmer subgroup; red arrows indicate poor adaptation and detachments; (c) interface of EverX airstream subgroup; the optimal integrity of the interface can be seen; (d) interface of Grandio control subgroup; (e) interface of Grandio warmer subgroup; arrows point to defects; (f) interface of Grandio airstream subgroup

DISCUSSION

This study assessed the effect of preheating of a nano-hybrid and a bulk-fill composite with a warm airstream on their MTBS to dentin. The results showed the lowest MTBS in the warmer subgroup and the highest in warm airstream subgroup in both composite types. The highest MTBS was recorded in EverX composite exposed to a warm airstream, followed by Grandio composite exposed to warm airstream. The MTBS of EverX composite was significantly higher than that of Grandio composite only in the warmer group; the difference in this regard was not significant in airstream and control subgroups.

To the best of the authors' knowledge, no previous study has evaluated the effect of preheating of composite with warm airstream on its MTBS to dentin. However, the effect of preheating with warmer has been previously investigated but mostly on the physical properties of the composite, and a number of studies on the effect of preheating of composites on their MTBS is limited. Only one study was found by Davari *et al*.,[9] on the effect of preheating of composite on its MTBS to dentin. However, they warmed the composite to 37°C. No previous study appears to be

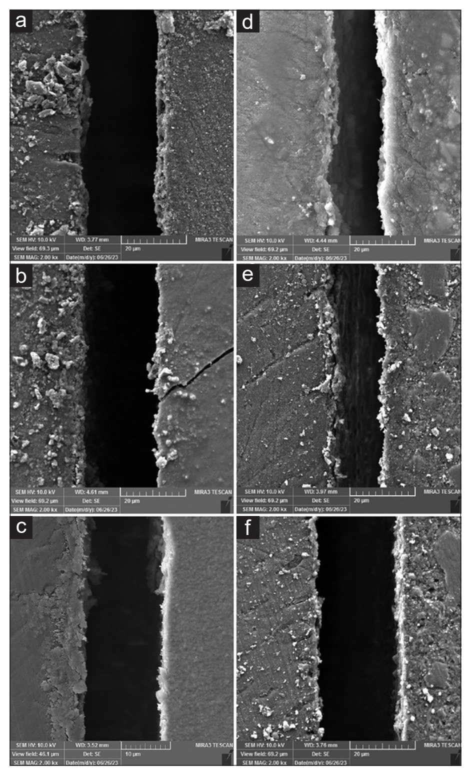

Figure 3: SEM micrographs at ×2000 magnification; (a) interface of EverX control subgroup; (b) interface of EverX warmer subgroup; (c) interface of EverX airstream subgroup; (d) interface of Grandio control subgroup; (e) interface of Grandio warmer subgroup; note the crack at the bonding interface to dentin; (f) interface of Grandio airstream subgroup

available on the effect of preheating of composite to 50°C on its MTBS to dentin. Görüş[10] showed that preheating of resin cement to 50°C decreased the MTBS of onlays fabricated by EverX Posterior base. Considering the low MTBS in the warmer subgroups in the present study, it appears that preheating of composite in a warmer at high temperatures may decrease its MTBS to dentin, despite improving the physical properties of the composite. Davari *et al*.[9] found that preheating of composite to 37°C improved the MTBS. Demirbuga *et al*.[11] demonstrated that preheating of composite to 68°C increased its micro-shear bond strength to dentin, compared with the control group. The problem related to the 30 s transfer time of composite from the warmer to the cavity before curing was nonexistent in the studies by Davari *et al*.,[9] and Demirbuga *et al*.,[11] because the composite specimens were placed on dentin models immediately after preheating and cured. In the present study, the occlusal surface of the teeth was reduced by 4 mm to ensure no enamel remaining; however, Davari *et al*.[9] and Demirbuga *et al*.[11] reduced the teeth by 2 mm. Deep dentin has larger dentinal tubule openings and a lower amount of intertubular dentin, than superficial dentin, which results in a lower bond strength to composite,[12] and may explain lower MTBS values obtained in the present study.

One major problem in preheating composite with warmer is the temperature drop in the process of composite transfer from the warmer into the cavity, its forming, and light curing. Lempel *et al*.[13] showed that the composite temperature dropped by 26°C in the process of delivery into the cavity, forming, and subsequent light-curing for 20 s, which results in tendency of monomers to return to the center and subsequent reduction of viscosity.

Preheating of composite increases the vibration and energy of monomers and can also enhance the collision frequency and affect the DC of composite.[14] Lempel *et al*.[13] evaluated the effect of temperature on DC of glass-fiber reinforced bulk-fill resin-based composite and sculptable conventional resin-based composite. They indicated that preheating of composite to 55°C significantly increased the DC of both composite types. Furthermore, the increase in DC was greater at the top surface, compared with the bottom surface of the composite. Tauböck *et al*.[15] evaluated the effect of preheating on DC and polymerization shrinkage of bulk-fill high-viscosity composite resins including Tetric EvoCeram Bulk Fill, X-tra fil, QuixFil, and SonicFill, as well as Tetric EvoCeram nano-hybrid composite. They showed that preheating to 68°C increased the DC of Tetric EvoCeram Bulk Fill but had no such effect on other composites. Preheating decreased the polymerization shrinkage of all composite types. They concluded that the type of composite and preheating both had a significant effect on polymerization shrinkage. However, the temperature drops in the process of delivery of composite from the warmer can affect the results and may explain inferior results obtained in the warmer subgroups in the present study.

Unlike the results of Tauböck *et al*.,[15] Daronch *et al*.[7] indicated that preheating of composite in a warmer increased the DC due to increased vibration of filler content and resin monomers; however, this increase would be associated with greater polymerization shrinkage and stress.[16] In the present study, stereomicroscopic and SEM assessments of the bonding interface revealed cracks, defects, and nonuniformity of the interface in the warmer subgroups, which may be related to increased stress due to heating, and affect the bond strength. However, such defects were not observed in airstream subgroups, because the temperature drop of composite does not occur in this method.

Daronch *et al*.[17] showed that depending on the magnitude of temperature drop, duration of transfer time (time lapse between removal from the warmer and curing), and filler content, preheating can have different effects on the bond strength of composite resins. The primary viscosity of composite, filler volume, and composition of matrix monomers can affect the adhesion of composite resins.[18] Furthermore, Loumprinis *et al*.[19] evaluated the effects of preheating at 23°C, 30°C, 37°C, 45°C, and 54°C on the adhesion and viscosity of Grandio, Clearfil Majesty Posterior, Venus Pearl, Ecosite Elements Pure, and Filtek Supreme. They reported that generally, higher temperatures increase the adhesion of composites; however, increased adhesion increases the risk of void formation, which can adversely affect the bond strength. Thus, adhesion to composite instrument might have also occurred in the warmer subgroups of the present study and could have decreased the MTBS. However, it does not occur in preheating with warm airstream because temperature drop and adhesion to composite instrument do not occur in this method. Elhejazi[20] suggested a 15 s interval between preheating and light-curing of composite. They added that excessive preheating increases polymerization shrinkage; thus, they suggested to lower the intensity of light-curing to prevent further temperature rise and control polymerization shrinkage. Such thermal alterations may be another reason for the lower MTBS in the warmer subgroups in the present study.

The present results showed that the MTBS of Grandio composite in all subgroups was lower than that of EverX composite. It has been reported that higher filler content of composite resins improves their properties. However, in low-filler composites, temperature rise adversely affects the composite properties.[21] Consistent with this statement, Davari *et al*.[9] showed greater improvement in bond strength by preheating of composite with higher filler content (packable Filtek P60), compared with nanohybrid Filtek Z250 with lower filler content. One possible reason may be the increase in flowability of composite with higher filler content after heating, which would increase its penetration and flow into less accessible areas. It appears that higher filler content results in better properties following preheating of composite. However, this statement was in contrast to the present results, since Grandio composite resin has 87 wt% filler content while this rate is 74.2 wt% in EverX. It appears that filler content is not the only factor affecting the bond strength, and resin matrix and filler type also play a role in this respect. EverX is a bulk-fill composite while Grandio is an increment-fill composite. Bulk-fill composites have lower amounts of free monomers than increment-fill composites after curing, which affects their final bond strength.[22] In the present study, EverX showed a higher MTBS than Grandio in the control subgroup, which further supports the abovementioned hypothesis. Furthermore, the type of monomers is different in Grandio and EverX, which can affect the results. Bis-GMA monomers have the highest viscosity and lowest flexibility and have a lower percentage in bulk-fill composites to improve their properties.[23] Furthermore, the highest percentage of DC belongs to TEGDMA > UDMA > Bis-EMA > Bis-GMA.[24] In addition to lower filler content and lower percentage of Bis-GMA in EverX, it has short fibers, which enhance its bond strength and mechanical properties.[10] The effect of heating on adhesion may be another explanation for this finding. Loumprinis *et al*.[19] showed that preheating to 54°C increased the adhesion of DMG and Filtek Supreme composite resins. Filler type and monomers affect the adhesion of composites as well. Composite resins containing bipolar monomers such as bis-GMA can increase the adhesion of composite to metal instruments.[25] However, this effect was only noted in the warmer subgroups, and not in the airstream subgroups. The filler content of Grandio is one reason for the lower MTBS of the warmer subgroup of this composite, compared with EverX. Some of the composites are probably procured in the process of transfer from the warmer to the cavity. SEM micrographs also confirmed this statement since mixed failures had a higher frequency in the warmer subgroups of both composite types in the present study due to temperature loss, and adhesion to the instrument.

Darabi *et al*.[26] evaluated the effect of preheating of composite to 68°C on marginal adaptation of class II restorations, and reported higher marginal adaptation in the preheating group, compared with the control group. Preheating decreases the viscosity and improves the flowability of composites due to the increased vibration of monomers as a result of their increased surface energy.[27] Thus, marginal adaptation improves given that the preheated composite is immediately applied in the cavity.[28] Similar results were reported by some other studies.[4,29,30] Filler content also plays a role in this regard, such that composite resins containing inorganic fillers have a higher viscosity, and the effect of preheating would be greater on them.[31] Nonetheless, the MTBS of the warmer subgroups was lower than that of control subgroups, and SEM micrographs did not show superior adaptation of composite at the interface in this group.

Evaluation of the effect of preheating of composite by warm airstream was the main strength of this study since this topic has not been previously evaluated in the literature. However, this study had an *in vitro* design, and the clinical environment cannot be perfectly simulated *in vitro*. Thus, the results should be generalized to the clinical setting with caution. Furthermore, only two types of composite resins were evaluated in this study. Future studies should focus on a broader range of composite types and brands. Furthermore, the effects of preheating of composite on its adhesion, sensitivity, and their interaction effect on bond strength should be investigated in future studies.

CONCLUSION

Preheating with a warm airstream significantly increased the MTBS of both composite types to dentin. Preheating with warmer to 50°C decreased the MTBS of both composite types, compared with the control group.

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Conflicts of interest

There are no conflicts of interest.

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