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RESEARCH AND EDUCATION

Thermal and bioactive optimization of a unidose 3-step etch-and-rinse dentin adhesive

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ABSTRACT

Statement of problem. The limited durability of resin-dentin bonds is considered a major disadvantage of adhesive restorations. Therefore, clinical strategies have been developed to improve hybrid layer stability over time. These strategies require testing.

Purpose. The purpose of this in vitro study was to evaluate the influence of preheating and the inclusion of a bioactive glass in a unidose 3-step etch-and-rinse adhesive system on the adhesion of direct composite resin restorations.

Material and methods. Dentin disks from 80 molars were assigned to 8 groups (n=10): CG-T1/CG-T2: control group; PG-T1/PG-T2: adhesive preheated to 68 °C; BG-T1/BG-T2: 0.05 mg of Bioglass 45S5 (BAG) (particle size: 3 μm) added to primer; PBG-T1/PBG-T2: adhesive and BAG-modified primer preheated to 68 °C. Sticks were fabricated for microtensile bond strength (μTBS) testing and were tested at 1 week (T1) and after 6 months (T2) of storage. μTBS data were analyzed by using 2-way ANOVA and the Tukey-Kramer post hoc test (α=.05). Scanning electron microscopy was used to analyze the failure mode. Attenuated total reflection Fourier transform infrared spectroscopy was used to quantitatively analyze the modifications to the chemical structure of the adhesive system from preheating and BAG inclusion.

Results. The mean bond strength values at 1 week were statistically different, with PG-T1 (69.8 ±7.8 MPa) superior to all other groups. CG-T1 (58.2 ±6.7 MPa), BG-T1 (60.7 ±4.4 MPa), and PBG-T1 (61.0 ±4.6 MPa) were not statistically different (P>.05). PG-T2 maintained the highest bond strength at 6 months (68.3 ±3.7 MPa), with no decrease in μTBS observed over time. Failure modes were mostly adhesive. Attenuated total reflection Fourier transform infrared spectroscopy analysis reported that primer preheating caused solvent evaporation and revealed that preheating the bonding agent promoted the condensation reaction between the silane and adhesive fillers.

Conclusions. No decrease in μTBS was observed for any group after 6 months. Preheating the adhesive system (primer and bonding resin) significantly increased the 1-week and 6-month μTBS. Inclusion of BAG did not affect the bond strength. (J Prosthet Dent 2020;■:■-■)

The decrease in dentin-resin bond strength is still a challenge in adhesive dentistry. To date, 3-step etch-and-rinse adhesive systems have been the most studied and reliable adhesives. A filled 3-step etch-and-rinse adhesive system (Optibond FL; Kerr Corp) seems to be the best representative of these adhesives, as it provides the highest microtensile bond strength (μTBS) and the greatest stability at 1 year.[1-3] One of the main tenets in dentin bond durability is the complete sealing of the demineralized collagen network by hydrophilic resin monomer infiltration.[4-7] An unprotected interface can lose its integrity because of water sorption–induced hydrolysis of the hydrophilic adhesive resin components[8] and through collagen fibril degradation by matrix metalloproteinases that are activated by acid etching.[9]

Remineralization techniques represent a new approach to improving the stability of the hybrid layer. The inclusion of bioactive elements in dentin may provide a progressive dehydration mechanism that replaces the extrafibrillar and intrafibrillar water and promotes the growth of hydroxyapatite crystals.[10] Different bonding techniques[11,12] and remineralization agents have been used to promote resin-dentin remineralization.[13,14]

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Clinical Implications

The preheating of restorative materials increases their physical and mechanical properties. Preheating an adhesive system improved silane reactivity and its interaction with adhesive filler compounds, resulting in higher bond strength values that remained stable after 6 months of specimen storage.

Nevertheless, the authors are unaware of attempts to include the gold standard bioactive material Bioglass 45S5 (BAG−45% SiO_2, 24.5% Na_2O, 24.5% CaO, 6% P_2O_2 -%wt)[15] into an existing commercial adhesive system.

A trend in restorative dentistry is to use preheated dental materials[16-20] to improve their conversion rate,[21] facilitate their placement,[22] increase their flow,[23-25] and enhance their bonding performance.[26,27] Simulating intraoral temperature is an important issue in validating in vitro studies; however, preheating etch-and-rinse and universal adhesives at 37 °C did not significantly modify dentin μTBS.[28,29] When 2-step etch-and-rinse adhesives were preheated at a higher temperature (50 °C), the μTBS values were affected by the vaporization temperature of their solvents, and acetone adhesive systems were affected because of the lower vaporization temperature of their solvent when compared with ethanol adhesive systems, which have a higher vaporization temperature.[28,30] Preheating logically requires the use of a sealed unidose system to prevent evaporation of the components. Preheating a filled 3-step etch-and-rinse adhesive system remains to be tested and could be significant in improving the seal of the hybrid layer by enhancing adhesive flowability and interdiffusion.

Therefore, the purpose of this in vitro study was to evaluate the influence of preheating and/or the inclusion of BAG in a unidose filled 3-step etch-and-rinse adhesive on dentin bond strength and the adhesive chemical compounds. The null hypotheses were that adhesive preheating and/or BAG inclusion would not improve the adhesion of direct restorations and that no difference would be found in μTBS after 6 months of specimen storage.

MATERIAL AND METHODS

On approval from the Ethical Review Committee of the University of Southern California, (protocol HS-17-00703), 80 extracted sound human molars were used. The molars were stored at 4 °C in thymol solution up to 1 month before use. Flat mid-coronal dentin surfaces were created by using a low-speed diamond saw (Isomet; Buehler Illinois Tool Works Inc). Any remaining enamel was removed by finishing with 600-grit SiC paper (Gatorgrit; Ali) under water. The teeth were assigned to 8 experimental groups (n=10) (Table 1).

Bonding procedures were done with a 3-step etch-and-rinse adhesive unidose system (Optibond FL; Kerr Corp). The dentin surfaces were treated with 35% phosphoric acid (Ultra Etch 35%; Ultradent Products, Inc) for 15 seconds and rinsed for 1 minute. After acid conditioning, the primer solution was lightly rubbed on the dentin for 30 seconds and air dried for 5 seconds. Adhesive was applied with a light brushing motion for 30 seconds and polymerized for 20 seconds at 1000 mW/cm^2 by using a light-emitting diode unit (VALO Curing Light; Ultradent Products, Inc). To reduce polymerization contraction stress, the specimens were restored by using 5 horizontal increments of composite resin of 1 mm in thickness (Filtek Z100; 3M) covering the entire dentin. Each layer was polymerized for 20 seconds at 1000 mW/cm^2 (VALO Curing Light; Ultradent Products, Inc). Considering that bioactive agents need time to react with mineral tissue and create a hydroxyapatite layer,[15] all restored specimens were stored in distilled water at room temperature for 1 week (T1) or 6 months (T2, water replaced weekly) before testing.

The dentin bonding agent mode of use was performed according to the experimental groups (Table 1). In CG-T1 and CG-T2, the adhesive system was used without any modification. For PG-T1 and PG-T2, the adhesive components (primer and resin, Optibond FL; Kerr Corp) were preheated for 15 minutes in a composite resin preheater (Calset; AdDent Inc) at 68 °C just before application. The adhesive system of BG-T1 and BG-T2 was modified by adding 0.05 mg of BAG[15] powder to the primer. In PBG-T1 and PBG-T2, the adhesive system received both modifications, its primer was modified by adding 0.05 mg of BAG, and both primer and bond components were preheated as previously indicated.

Each restored specimen was sectioned in an occlusal-apical direction through the tooth-restoration interface to produce approximately 0.9-mm-thick slices. Each slice was sectioned in a mesial-distal direction to obtain dentin-restoration sticks with a cross-sectional area of approximately 0.9 mm^2, obtaining 10 specimens per tooth. Fractured surfaces were measured by using a digital caliper (Zaas Precision; Amatools Co). The resin-dentin sticks were attached to the grips of a testing jig by using cyanoacrylate cement (Super Bonder; Henkel Loctite Corp) and were tested in a microtensile tester machine (Bisco Inc) at a crosshead speed of 53 N per minute until failure. The maximum load at fracture (N) and the cross-sectional area of each failed stick was used to calculate μTBS values in MPa.[31]

The mean microtensile bond strength from the 10 sticks was used as a single measurement, yielding 10 measurements per group. Statistical analyses were

Table 1. Experimental groups and mode of use of adhesive system

	Groups							
Experimental variables	**CG-T1**	**CG-T2**	**PG-T1**	**PG-T2**	**BG-T1**	**BG-T2**	**PBG-T1**	**PBG-T2**
Preheating for 15 min/68 °C	No	No	Yes	Yes	No	No	Yes	Yes
Addition of 0.05 mg of Bioglass 45S5 to primer	No	No	No	No	Yes	Yes	Yes	Yes
Delay before μTBS testing	1 week	6 months	1 week	6 months	1 week	6 months	1 week	6 months

CG-T1, control group tested after 1 week; CG-T2, control group tested after 6 months, PG-T1, group with preheated adhesive tested after 1 week; PG-T2, group with preheated adhesive tested after 6 months, BG-T1, group with Bioglass 45S5 added to primer tested after 1 week; BG-T2, group with Bioglass 45S5 added to primer tested after 6 months; PBG-T1, group with preheated adhesive and Bioglass 45S5 added to primer tested after 1 week; PBG-T2, group with preheated adhesive and Bioglass 45S5 added to primer tested after 6 months.

Table 2. Microtensile bond strength values (MPa) according to experimental group

	Groups							
Experimental results	**CG-T1**	**CG-T2**	**PG-T1**	**PG-T2**	**BG-T1**	**BG-T2**	**PBG-T1**	**PBG-T2**
μTBS results	58.2 ±6.7 A	60.5 ±5.3 A	69.8 ±7.7 B	68.3 ±3.7 B	60.7 ±4.4 A	64.0 ±3.2 AB	61.0 ±4.6 A	65.5 ±6.5 AB
Premature failures/tested sticks	(5/100)	(3/100)	(3/100)	(3/100)	(8/100)	(6/100)	(4/100)	(8/100)

First row indicates μTBS mean ±standard deviation in MPa, different uppercase letters indicate significant statistical difference ($P<.05$). Second row reports pretesting failures and number of sticks tested. μTBS, microtensile bond strength; CG-T1, control group tested after 1 week; CG-T2, control group tested after 6 months, PG-T1, group with preheated adhesive tested after 1 week; PG-T2, group with preheated adhesive tested after 6 months, BG-T1, group with Bioglass 45S5 added to primer tested after 1 week; BG-T2, group with Bioglass 45S5 added to primer tested after 6 months; PBG-T1, group with preheated adhesive and Bioglass 45S5 added to primer tested after 1 week; PBG-T2, group with preheated adhesive and Bioglass 45S5 added to primer tested after 6 months.

performed by using statistical software (R statistical software R i386 v3.0.2; R Foundation for Statistical Computing). Data normality was confirmed by using the Shapiro-Wilk test ($P=.735$), and the homogeneity of variances was determined by using the Bartlett test ($P=.127$). Bond strength data obtained from the 8 experimental groups were analyzed by using 2-way ANOVA (dentin bonding strategy and storage time) and the Tukey-Kramer post hoc test ($\alpha=.05$) with statistical software (R statistical software R i386 v3.0.2; R Foundation for Statistical Computing).

The failure modes of 5 fractured sticks from each tooth were evaluated by using a scanning electron microscope (Superscan SS-550; Shimadzu Corp) at ×100 magnification. The fractured surfaces were placed on aluminum disks and sputter-coated with a gold-palladium alloy (Bal-Tec; Capovani Brothers Inc). Failure patterns were classified in the following categories[32]: adhesive failure when the fracture site was located between the adhesive and dentin; mixed failure if the fracture involved the dentin-adhesive interface, including cohesive failure of dentin and composite resin; or cohesive failure in the substrate (underlying dentin or overlying composite resin). Chi-square analyses were performed to compare the failure modes in all the groups (Excel; Microsoft Corp).

For the molecular composition analysis, attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) analysis was performed by using an FTIR spectrophotometer (Bruker Optik GmbH) equipped with a diamond ATR accessory. The chemical structures of the control, preheated, BAG-modified, and preheated BAG-modified primer solutions, the control, and preheated adhesive solutions were analyzed. Adhesive specimen disks were prepared (n=5) by polymerizing 1.8 mL of control and preheated adhesive on a polyester strip for 20 seconds at 1000 mW/cm^2 (VALO Curing Light; Ultradent Products, Inc). Spectra of the primer specimens were obtained by dropping 1.8 mL of each primer into a concave ATR-FTIR device. The ATR-FTIR spectra from the BAG powder was also obtained. Spectra were collected in the range of 4000 to 600 cm^{-1} at 4 cm^{-1} resolution for a total of 128 scans. All spectra were submitted to baseline correction. Visual qualitative analysis was done for each spectra range to identify possible chemical modifications in the primer and the adhesive compounds. For this purpose, the spectra of pure primer and pure adhesive were used as controls.

RESULTS

μTBS results are presented in Table 2. The 2-way ANOVA (Table 3) indicated a significant effect for the dentin bonding strategy but not the storage time. The interaction term was not significant. The preheated group (PG-T1) provided significantly stronger bond strength when compared with the other groups. CG-T1, BG-T1, and PBG-T1 did not differ from each other. PG-T2 maintained the highest bond strength at 6 months, and no decrease in μTBS was observed for any group after 6 months.

The failure mode results, determined by scanning electron microscope, are presented in Table 4. Chi-square analysis was not significant. Most failures in all tested groups were adhesive failures. The types of failures occurring in the experimental groups are illustrated in Figure 1.

Table 3. Two-way ANOVA

Source	*df*	Type III Sum of Squares	Mean Square	F	*P*
Adhesive system modification	3	984.96	328.32	10.63	<.001
Storage time	1	96.97	96.97	3.14	.081
Interaction	3	105.29	35.09	1.13	.340

P<.05 indicates significant difference. df, degrees of freedom; F, F-statistic.

Table 4. Failure mode according to experimental group

Failure modes	Groups							
	CG-T1	CG-T2	PG-T1	PG-T2	BG-T1	BG-T2	PBG-T1	PBG-T2
	Frequency (%)							
Adhesive	33 (66)	31 (62)	35 (70)	35 (70)	31 (62)	35 (70)	25 (50)	36 (72)
Mixed	12 (24)	18 (36)	11 (22)	14 (28)	15 (30)	13 (26)	22 (44)	12 (24)
Cohesive	5 (10)	1 (2)	4 (8)	1 (2)	4 (8)	2 (4)	3 (6)	2 (4)

μTBS failure modes , n=100 sticks for each group. No significant difference in failure mode between experimental groups. CG-T1, control group tested after 1 week; CG-T2, control group tested after 6 months, PG-T1, group with preheated adhesive tested after 1 week; PG-T2, group with preheated adhesive tested after 6 months, BG-T1, group with Bioglass 45S5 added to primer tested after 1 week; BG-T2, group with Bioglass 45S5 added to primer tested after 6 months; PBG-T1, group with preheated adhesive and Bioglass 45S5 added to primer tested after 1 week; PBG-T2, group with preheated adhesive and Bioglass 45S5 added to primer tested after 6 months.

The mean ATR-FTIR spectra for all the experimental conditions of primer and bonding agent solutions are shown in Figure 2. Primer preheating resulted in the presence of 2 new peaks, one at 1230 cm^{-1} (OH, aromatic ether)[33] and the other at 1125 cm^{-1} (C-O stretch, secondary alcohol).[33] When Bioglass was included in the primer solution at room temperature, its compounds were evidenced in the primer spectra in the wavelength range of 920 cm^{-1} to 840 cm^{-1} (PO_4 ν3 antisymmetric stretching mode).[34] These alterations were not found in the preheated primer with Bioglass. Preheating the adhesive resin increased intensity in the wavelength range of 1210 cm^{-1} to 850 cm^{-1} (peaks attributed to Si-O vibrations).[35-37]

DISCUSSION

The results of the present study led to the partial rejection of the first null hypothesis because the preheated specimens (PG-T1/PG-T2) presented significantly higher μTBS results when compared with the control (CG-T1/CG-T2) and remineralization groups (BG-T1/BG-T2 and PBG-T1/PBG-T2). The second null hypothesis was not rejected because storage time did not have an effect on μTBS. Chemical analysis supported the μTBS results because the preheating treatment modified the adhesive and primer solutions and improved their interactions with dentin.

Loguercio et al[28] reported that preheating an ethanol and water 2-step etch-and-rinse adhesive at 50 °C improved the quality of the hybrid layer by increasing the adhesive penetration and reducing the thickness of the adhesive layer. However, the use of a preheated solution did not affect either the adhesive degree of conversion or resin-dentin μTBS.[28,30] In the present study, preheating an ethanol 3-step etch-and-rinse filled adhesive system at a higher temperature (68 °C) caused a significant increase in resin-dentin μTBS. This difference may have been because of the difference in the preheated temperature tested, the adhesive system strategy, the duration of the preheating treatment, and the use of sealed unidose containers.

Adhesive solvent evaporation is a critical clinical step. When solvent evaporation is too fast, the monomer infiltration is affected, decreasing bond strength values.[38] However, if solvent remains in the adhesive layer, it can compromise the structural integrity of the hybrid layer.[39,40] Ethanol is the principal solvent of the tested adhesive system, and as its vaporization temperature is 78.3 °C,[16] preheating to 68 °C was effective in partly evaporating the ethanol without removing it completely.[20,38] The use of a sealed unidose adhesive system is pivotal to the preheating technique. The small single-use rocket-shaped containers of primer and adhesive of the Optibond FL system can be easily stored in the heating device and are completely sealed. When compared with a bottle system,[30] the unidose system prevents repetitive material heating that can modify the adhesive solvent composition.

Previous studies have preheated the primer and adhesive solution for 2 hours before its use[28,30]; however, according to the advice of the manufacturer, it is safe to preheat the primer for up to 20 minutes and the adhesive for less than 2 hours before application. Preheated materials, including restorative composite resins, can lose heat rapidly during the transfer to the mouth.[21] The temperature drop should be minimized by reducing the time between removal from the heating device and application in the mouth. When considering that the temperature of primer and adhesive drops during transfer and application, preheating should not have a negative effect on the pulp.[21]

The present experiment reported that the addition of bioactive particles to the primer did not affect the 6 months μTBS of the 3-step etch-and-rinse adhesive, even when combined with preheating. The remineralization process requires an exchange of ions (Na^+, Ca^{2+}, PO_4^{3-}, F^-) from the Bioglass silicate network to the surrounding dentin fluid.[12,14] This process induces calcium phosphate (CaP) precipitation and its subsequent crystallization into hydroxyapatite at the tissue surface.[13,15] This process produces electrostatic, ionic, and/or hydrogen bonding between the demineralized collagen and Bioglass silanols, which in turn may reduce collagen degradation.[10,12] The limited μTBS improvement in the present study might be explained by encapsulation of the bioactive glass in the resin network and insufficient storage time.

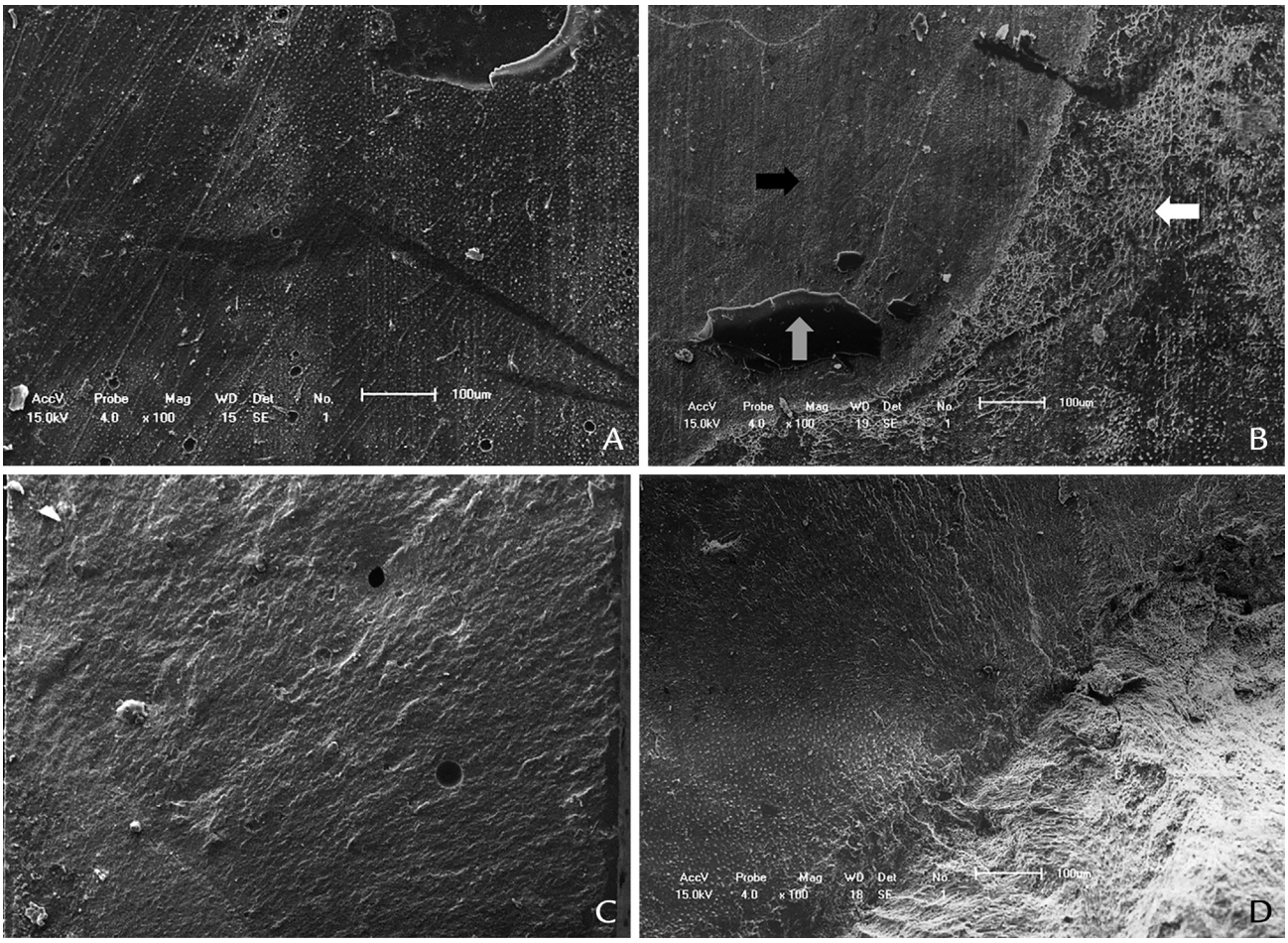

Figure 1. Scanning electron photomicrographs of microtensile specimens (Original magnification ×100). A, Adhesive failure in hybrid layer. B, Mixed failure in hybrid layer (*black arrow*), cohesive at dentin surface (*white arrow*), and cohesive at adhesive system (*gray arrow*). C, Cohesive failure at composite resin. D, Cohesive failure at dentin surface.

The findings of ATR-FTIR analysis revealed that preheating influenced the hydrophilic (primer) and hydrophobic (adhesive resin) parts of the adhesive and thus confirmed the μTBS results. The evaporation of primer solvents has been reported to depend on the number of hydrogen bonding sites between the polymer and the solvent. It is harder to volatize the solvent when there are many hydrogen bonds.[41] The appearance of the new peaks attributed to ether (1230 cm^{-1})[33] and secondary alcohol (1125 cm^{-1})[33] may be related to an alcohol intramolecular dehydration, suggesting an increase in alcohol solvent evaporation when the primer was submitted to a higher temperature.[38] A contribution of BAG phosphate compounds (920 cm^{-1} to 840 cm^{-1})[34] was evidenced in the spectra of the BAG modified primer, although this modification was not found in the preheated BAG modified primer.

The tested adhesive system is filled with a silanated barium aluminoborosilicate glass filler. Silanization of filler particles enhances the affinity of the inorganic particles with the resin monomers, improving the physical and mechanical properties of composite resins.[27] Silane heat treatment has been shown to enhance the bonding performance of silanized ceramic restorations and makes the condensation reaction more effective because it promotes the formation of a covalent bond between the silica and silane.[14,35] In addition, silane heat treatment as appears to reduce its hydrophilic constituents and consequently improve the bonding between composite resin and zirconia ceramic.[42] In the present study, the preheating treatment of the bonding agent modified the ATR-FTIR spectra range attributed to silanized SiO_2 particles (1210 cm^{-1} to 850 cm^{-1}),[43] indicating an increase in its silane hydrolysis and a consequent improvement in the condensation reaction between silane and the adhesive fillers.[27,42] From the ART-FTIR findings, the higher bond strength values found when adhesive was preheated can be

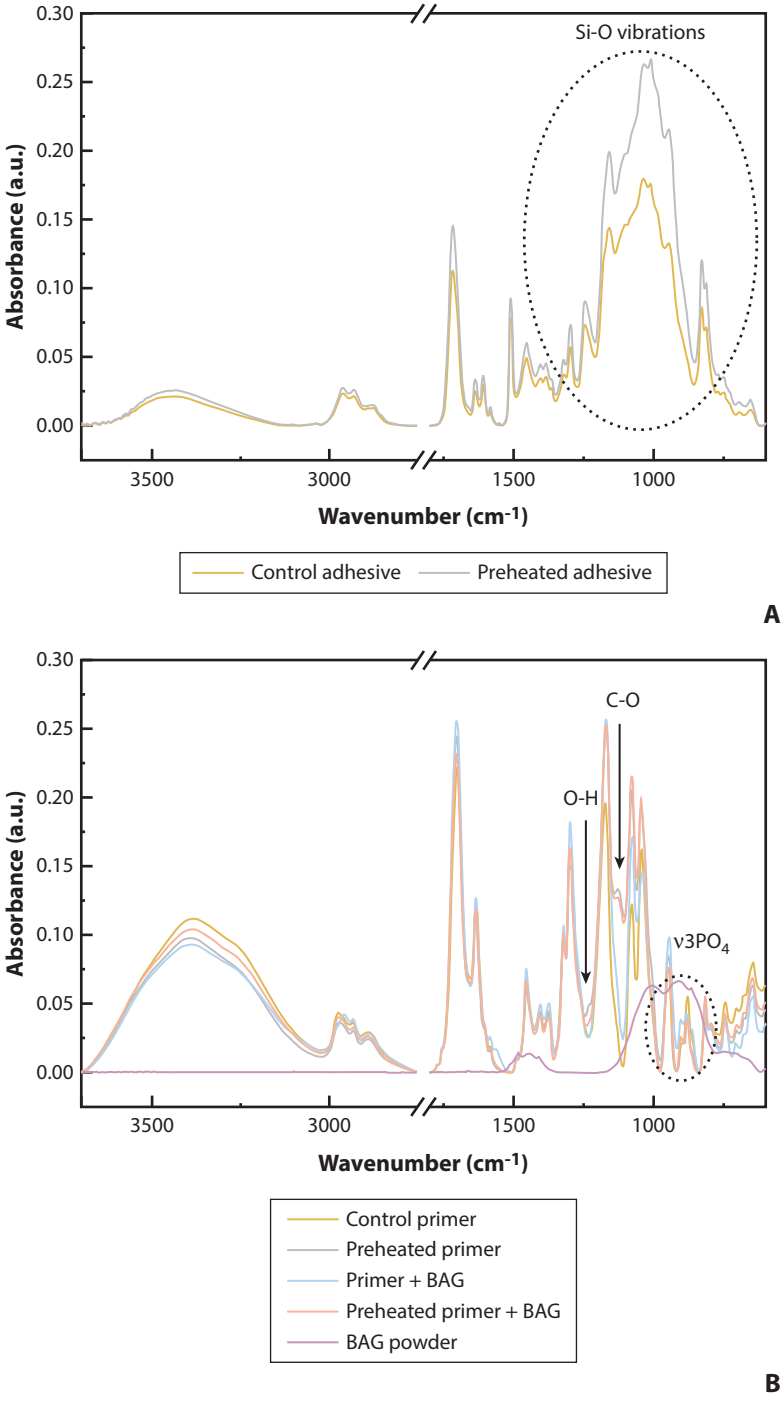

Figure 2. ATR-FTIR spectra of different modes of use, A, Bonding agent, B, Primer. ATR-FTIR, Attenuated total reflection Fourier transform infrared spectroscopy.

attributed to the chemical modifications to primer and adhesive compounds.

Limitations of the present study include its in vitro design by using extracted teeth. Dentin temperature and humidity seem to influence the μTBS results; additionally, adhesive permeability into dentin tissue can be modified when the connection with the pulp tissue is lost. These limitations could have been minimized if the teeth had been restored before their extraction. Further research is indicated to determine the best strategy (sequence, method) for the application of BAG to benefit a 3-step total etch-and-rinse adhesive. The high values of all μTBS at 6 months is encouraging and calls for further analysis of the stability of those values.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Preheating of an unidose filled 3-step etch-and-rinse adhesive resulted in significantly higher 1-week and 6-month μTBS.
2. Inclusion of BAG in the adhesive primer component, both at room and preheated temperature did not yield an improvement in μTBS.
3. None of the groups reported a decrease in μTBS values after 6 months of storage.
4. Adhesive system preheating induced primer solvent evaporation and improved the condensation reaction between silane and bonding agent fillers.

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