Original Article

An *In-vitro* Evaluation of the Shear Bond Strength to Dentin and Microhardness of Different Restorative Materials Placed by Different Techniques

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Abstract

Background: The mentioned advantages of bulk-fill composite resin and high-viscosity glass ionomer restorative materials have increased their use in restorative dentistry in recent years; accordingly, the bonding of these materials to dental tissues and their mechanical properties have become more important. **Aim:** This study aimed to evaluate the effects of different application methods on Vicker's hardness and shear bond strength of three different restorative materials. **Methods:** In this *in-vitro* study; Teflon molds were used for the microhardness test. In the control group, reinforced high-viscosity glass ionomer, high-viscosity glass ionomer, and flowable bulk-fill composite resin were applied by the manufacturer's instructions. In other groups, preheating, ultrasonic activation, and both preheating and ultrasonic activation were applied, respectively (n = 14). Microhardness values of the upper surfaces of the specimens were measured with Vicker's hardness measuring device. For the shear bond strength test, 84 intact human molar teeth were used. The teeth were sectioned two in the mesiodistal direction (n = 14). The materials were applied to the dentin using the same placement protocols as those used in the hardness test. After the specimens were maintained at 37°C for 24 h, the shear bond strength test was performed using a universal test device. **Statistical Analysis Used:** The data were analyzed using SPSS 26.0 at a 95% confidence level. The Mann–Whitney test was also used for the statistical analysis of the data (P = 0.05). **Results:** Preheating the restorative materials significantly decreased the shear bond strength in the flowable bulk-fill composite resin group (11.77 ± 4.46 MPa) compared with that in the control group (12.14 ± 4.23 MPa) ($P < 0.05$) but significantly increased the shear bond strength in the reinforced high-viscosity glass ionomer group (3.91 ± 2.93 MPa) ($P < 0.05$). **Conclusions:** It can be concluded that preheating before application may increase the shear bond strength of reinforced high-viscosity glass ionomer.

Keywords: *Bulk-fill, composite resin, glass ionomer, heat, ultrasonic activation*

Introduction

Currently, the most preferred material in restorative dentistry is composite resin. However, composite resins have disadvantages such as polymerization shrinkage, the need for serious technical precision during application, and long processing time owing to the requirement for layered application.[1] Various composite resin materials have been produced by updating the material structure to overcome these disadvantages. One of these composite resins is bulk-fill composite resins.

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Glass ionomers have also started to be used as permanent restorative materials.[2] However, the lack of sufficient physical and mechanical properties of glass ionomers

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limits their use as permanent restorative materials.[3] However, a series of updates have been made in the structure and application of glass ionomer materials to overcome such a situation. Among these updates are the addition of different molecules to strengthen the material structure, the addition of different structures to improve the antibacterial properties, and the use of application techniques such as heating, and ultrasonic activation.

Some of the advantages of preheating resin-containing materials include increasing the degree of conversion, improving the marginal adaptation of restorations by decreasing viscosity, and reducing polymerization shrinkage.[4] This technique also increases the rate of conversion of monomers to polymers and microhardness without accelerating the time at which maximum polymerization is achieved. This improvement is probably attributed to the increased molecular mobility and collision frequency of reactive molecules.[5]

Heat curing is a relatively new technique that uses radiant heat to accelerate the curing reaction of conventional glass ionomer cement (GIC). This technique helps overcome the early moisture sensitivity problem of GIC. Several researchers have investigated the effects of thermosetting on the mechanical properties of various GIC and reported an increase in surface microhardness and flexural strength.[6]

Heat treatment increases the mobility of both polymer segments and reactive free radicals formed during polymerization, which consequently increases the degree of conversion of monomers to polymers and allows the crosslinking of polymers to increase.[7]

It has been reported that GICs, whose hardening is completed by applying ultrasonic energy, show higher microhardness than do conventional GICs.[8] The increase in microhardness is explained by the fact that ultrasonic waves cause closer mixing of acid and powder, creating more contact area between acid and glass. Ultrasonic waves reduce the size of particles in the glass phase and cause more reaction surfaces with acid, resulting in a more compact solid with a better arrangement of residual glass particles.[9] In ultrasonic wave activation, GICs are preferred in terms of longer life and more resistance to forces.[10]

The mentioned advantages of bulk-fill composite resin and high-viscosity glass ionomer restorative (HVGIC) materials have increased their use in restorative dentistry in recent years; accordingly, the bonding of these materials to dental tissues and their mechanical properties have become more important.[11]

Dionysopoulos *et al*.[12] evaluated the effect of heating and ultrasonic activation on the microhardness of GIC materials and reported that both techniques increased the surface microhardness. The application of heat to the GIC surface increased the ion mobility in the first stage of hardening, reduced the viscosity, and accelerated and improved the curing reaction. Conversely, ultrasonic activation increased the rate of the curing reaction depending on the kinetic energy and temperature increase (thermocatalysis). In another study, it was reported that ultrasonic activation under similar experimental conditions caused a temperature increase of approximately 13°C on the surface of GIC materials.[13]

Unlike resin-containing materials, GICs show increased viscosity when heated. The application of heat is believed to increase the rate of ion diffusion, speed up the reaction, and reduce the working and setting times. However, after GICs are mixed, heating improves the physical and chemical properties of the materials.[5] It has been reported that heat application can increase the surface microhardness, abrasion resistance, bonding to dental tissues, and compressive strength of glass ionomer materials and reduce water absorption, solubility, and microleakage.[14] A sufficient surface microhardness of a restorative material can increase its resistance to high chewing forces and reduce marginal microleakage.[15] In the study by Dehurtevent *et al*.,[16] heating and ultrasonic activation and a combination of both were applied to reinforce high-viscosity GICs, and the mechanical properties were evaluated. Both heating and ultrasonic activation improved the surface microhardness of the materials after 24 h.

There are a limited number of studies in which different methods are applied to improve the physical properties of RHVGIC materials and bulk-fill composite resins. However, within our limited research, the effect of using both preheating and ultrasonic activation, two HVGIC materials with different contents and flowable bulk-fill (FBF) composite resin materials have not been evaluated on the shear bond strength to dentin and microhardness. In this study, the application of reinforced high-viscosity glass ionomer restorative material (RHVGIC), HVGIC, and FBF composite resin restorative material was evaluated for the effects on shear bond strength and Vicker's hardness after different application methods. Unlike the literature, this study aimed to evaluate the effects of preheating at different times and temperatures, ultrasonic activation at different times, and the combination of both techniques on the shear bond strength to dentine and Vicker's hardness of the materials.

Material and Methods

The materials used in the study and their contents and application methods are shown in Table 1, while the

experimental groups and the procedures applied to the specimens are displayed in Table 2.

Vicker's hardness test

Teflon molds with a depth of 4 mm and a diameter of 4 mm were used for Vicker's hardness test. During the preparation of each specimen, the restorative materials were applied based on the groups by placing transparent tape on top of the cement glass at the bottom and a Teflon mold on top. The procedures applied according to the experimental groups are shown in Table 2. Thereafter, a transparent tape was placed on the applied restorative material, and a cement glass was placed on top. A slight force was then applied to obtain a flat surface.

The hardness of the upper surfaces of the prepared specimens was measured using a Vicker's hardness tester (HMV-700 Micro Hardness Tester, Shimadzu, Japan). During the test, the device was operated by applying a load of 100 g, equivalent to 980.7 mN, for 10 s. The rhombic projection corners formed were then marked under the ocular at ×40 magnification, and Vicker's hardness was measured. The measurements were repeated at three different points for each specimen. The average of the measured values was recorded as the surface hardness.

Shear bond strength test

Eighty-four intact human molar teeth with extraction indications were included in the study. The teeth were divided into two in the mesiodistal direction using a slowly rotating diamond cutter. The buccal/palatal–lingual surfaces of the teeth were placed on the upper side and in pink cold acrylic surrounded by a polyvinylchloride cylinder. The enamel surface was abraded using a 300-rpm sanding and polishing device (Mecapol P230 Press, France) under water cooling until the dentin surface was exposed using an 800-grit sandpaper. The specimens were randomly divided into 12 groups (n = 14). The List Box method in Microsoft Excel was used to randomize the specimens.

The materials were placed on the dentin surface using a transparent plastic tube (4 mm in depth and 2.72 mm in diameter) using the placement techniques shown in Table 2.

The specimens were kept in distilled water for 24 h in an oven at 36.5°C. After 24 h, the shear bond strength test was performed using the Universal Tester (Schimadzu IG-IS, Kyoto, Japan) at a speed of 1 mm/min. The Newton values obtained were converted to megapascal values by calculating the surface area of the specimens, and the obtained data were recorded for statistical analysis.

Fracture type analysis

The surface where refraction occurred in all specimens was examined using a stereomicroscope (Olympus SZ-40, Tokyo, Japan) at ×40 magnification. The fracture types were determined by the following classification, and the data obtained were recorded.

- *Adhesive:* 75% or more of the fracture is between the tooth and the restorative material.
- *Cohesive:* 75% or more of the fracture is in the restorative material or the tooth itself.
- *Mixed:* Mixed failure is observed in adhesive–restorative material–tooth tissue.

Statistical analysis

The data were analyzed using SPSS 26.0 at a 95% confidence level. The Mann–Whitney test was also used for the statistical analysis of the data ($P = 0.05$).

Results

Vicker's hardness

The average Vicker's hardness was the highest in Group 3c (73.82), Group 3b (68.24), and Group 3 (52.15) and lowest in Group 2a (34.47), Group 1a (36.17), Group 1b (39.68), and Group 2b (40.32) [Table 3]. The *P* values calculated using the Mann–Whitney test for the intra-group comparison of Vicker's hardness are shown in Table 4. The distribution of the average Vicker's hardness by group is shown in Figure 1.

The FBF group showed the highest Vicker's hardness. According to the Mann–Whitney test, no significant difference was found in Vicker's hardness between the groups in which RHVGIC and HVGIC were applied with the same techniques. The group in which FBF was placed with both heating and ultrasonic activation showed a significantly higher Vicker's hardness than did both the control and heat-applied groups. There was no significant difference between the group in which FBF was placed with heating and the group in which RHVGIC was placed with both heating and ultrasonic activation. The group in which HVGIC was placed with ultrasonic activation showed a significantly higher Vicker's hardness than did the group in which the material was placed with heating.

Shear bond strength

The average shear bond strength was the highest in Group 3 (19.93), Group 3b (19.37), Group 3a (17.25), and Group 3c (16.54) and lowest in Group 1 (5.07), Group 2 (5.79), Group 1b (6.64), Group 1c (5.91), and Group 2c (5.56) [Table 5]. The *P* values calculated using the Mann–Whitney test for the within-group comparison of the shear bond strength are displayed in Table 6. The distribution of

Table 1: Materials used in the study, contents and application methods

Material Name	Material Type	Manufacturer	Contents	Method of Application
Equia Forte HT	Reinforced High-Viscosity Glass Ionomer Restorative Material	GC Corp., Tokyo, Japan	Powder: Fluoroaluminosilicate glass, polyacrylic acid, iron oxide Liquid: Polybasic carboxylic acid, water	• It was mixed in an amalgamator for 10 seconds.
Fuji IX GP	High-Viscosity Glass Ionomer Restorative Material	GC Corp., Tokyo, Japan	Powder: Aluminosilicate glass, polyacrylic acid Liquid: Polyacrylic acid, water	• It was mixed in an amalgamator for 10 seconds.
Estelite Bulk-Fill Flow	Flowable Bulk-Fill Composite Resin	Tokuyama Dental Corp., Tokyo, Japan	Filler content: 56% by volume, 70% by weight New organic inorganic hybrid filler, supra nano spherical filler (SiO2-ZrOs), Bis-GMA, TEGDMA, BisMPEPP, CQ, Radical-Amplified Photopolymerization initiator	• It was applied in a layer of 4 mm. • It was polymerized with LED light device for 10 seconds.
Equia Forte Coat	Coating Agent	GC Corp., Tokyo, Japan	Methylmethacrylate, multifunctional methacrylate, camphorquinone	• After being applied to the material surface, it was polymerized for 20 seconds with an LED light device.
G - Premio Bond	Universal Bonding Agent	GC Corp., Tokyo, Japan	Acetone (25-50%), 2-hydroxy-1,3 dimethacryloxypropane (10-20%), methacryloyloxydecyl dihydrogen phosphate (5-10%), 2,2-ethylenedioxydiethyl dimethacrylate (1 5%), diphenyl (2,4),6 trimethylbenzoyl-phosphine oxide (1–5%), 2,6-di-tert-butyl-p cresol (<0.5%)	• Applied to the dentin surface. • After waiting for 10 seconds, it was air-dried for 5 seconds. • It was polymerized with LED light device for 10 seconds.
Cavity Conditioner	Cavity Conditioner	GC Corp., Tokyo, Japan	20% Polyalkenoic acid, 3% aluminum chloride (pH 1.2)	• It was applied to the dentin surface for 10 seconds. • Washed with water and rinsed gently.

Table 2: Experimental groups and procedures applied to specimens

Experimental Groups	Procedures Applied to Specimens
Group 1	Reinforced High-Viscosity Glass Ionomer Restorative Material (Control)
Group 1a	Reinforced High-Viscosity Glass Ionomer Restorative Material immersed in water at 50°C for 1 min
Group 1b	Reinforced High-Viscosity Glass Ionomer Restorative Material with 5 s ultrasonic activation with cavitron during placement
Group 1c	Reinforced High-Viscosity Glass Ionomer Restorative Material, which is kept in water at 50°C for 1 min and ultrasonic activation is applied with a cavitron for 5 s during placement.
Group 2	High-Viscosity Glass Ionomer Restorative Material (Control)
Group 2a	High-Viscosity Glass Ionomer Restorative Material, which is kept in water at 50°C for 1 min
Group 2b	High-Viscosity Glass Ionomer Restorative Material, which is ultrasonically activated with a cavitron for 5 s during placement
Group 2c	High-Viscosity Glass Ionomer Restorative Material, which is kept in water at 50°C for 1 min and ultrasonic activation is applied with a cavitron for 5 s during placement.
Group 3	Flowable Bulk-fill Composite Resin (Control)
Group 3a	Flowable Bulk-fill Composite Resin, which is kept in water at 50°C for 1 min
Group 3b	Flowable Bulk-fill Composite Resin with 5 s ultrasonic activation with cavitron during placement
Group 3c	Flowable Bulk-fill Composite Resin, which is kept in water at 50°C for 1 min and ultrasonically activated for 5 s with a cavitron during placement.

the average shear bond strength by group is shown in Figure 2.

FBF was the material with the highest shear bond strength. The shear bond strength significantly differed in all groups in which RHVGIC and HVGIC were applied. However, the shear bond strength was significantly lower in the heat-applied group than in the control group.

No significant difference was found in the shear bond strength between the groups in which RHVGIC and

Table 3: Descriptive statistics of Vicker's Hardness measurements by groups

Vicker's Hardness Groups	Mean (Standard Deviation)
Group 1	45.47 (12.62)
Group 1a	36.17 (4.49)
Group 1b	39.68 (4.83)
Group 1c	45.50 (18.98)
Group 2	41.44 (10.85)
Group 2a	34.47 (4.94)
Group 2b	40.32 (5.45)
Group 2c	40.98 (11.06)
Group 3	52.15 (6.03)
Group 3a	48.57 (2.01)
Group 3b	68.24 (18.09)
Group 3c	73.82 (31.00)

Table 4: Comparison of Vicker's hardness measurements by groups

	Group 1	Group 2	Group 3	Group 1a	Group 2a	Group 3a	Group 1b	Group 2b	Group 3b	Group 1c	Group 2c	Group 3c
Group 1	X	0.168	0.183	0.108	0.031*	0.491	0.408	0.927	0.002*	0.613	0.679	0.003*
Group 2	X	X	0.024*	0.581	0.077	0.154	0.927	0.909	0.000*	0.800	0.800	0.000*
Group 3	X	X	X	0.000*	0.000*	0.129	0.000*	0.000*	0.001*	0.022*	0.001*	0.000*
Group 1a	X	X	X	X	0.370	0.000*	0.069	0.029*	0.000*	0.421	0.215	0.000*
Group 2a	X	X	X	X	X	0.000*	0.010*	0.015*	0.000*	0.069	0.118	0.000*
Group 3a	X	X	X	X	X	X	0.000*	0.000*	0.000*	0.054	0.002*	0.000*
Group 1b	X	X	X	X	X	X	X	0.927	0.000*	0.890	0.550	0.000*
Group 2b	X	X	X	X	X	X	X	X	0.000*	0.713	0.291	0.000*
Group 3b	X	X	X	X	X	X	X	X	X	0.001*	0.000*	0.748
Group 1c	X	X	X	X	X	X	X	X	X	X	0.927	0.002*
Group 2c	X	X	X	X	X	X	X	X	X	X	X	0.000*
Group 3c	X	X	X	X	X	X	X	X	X	X	X	X

*P<0.05 significant difference, P>0.05 no significant difference; Mann Whitney

Table 5: Descriptive statistics of shear bond strength measures by groups

Shear Bond Strength Groups	Minimum (Standard Deviation)
Group 1	3.10 (1.23)
Group 1a	3.91 (2.93)
Group 1b	2.68 (3.05)
Group 1c	3.15 (1.61)
Group 2	2.25 (2.69)
Group 2a	3.52 (2.87)
Group 2b	2.53 (3.34)
Group 2c	2.68 (2.76)
Group 3	12.14 (4.23)
Group 3a	11.77 (4.46)
Group 3b	12.14 (4.35)
Group 3c	12.11 (3.47)

HVGIC were placed with the same techniques. The shear bond strength in the group in which RHVGIC was placed with heating was significantly higher than that in the control group.

Fracture type

In Group 2, mixed-type fracture was observed in 92.9% (13 specimens) of the specimens, and adhesive-type fracture was observed in 7.1% (1 specimen). In Group 3, mixed-type fracture was observed in 78.6% (11 specimens) of the specimens, and adhesive-type fracture was observed in 21.4% (3 specimens). Mixed-type fracture was observed in all specimens in Group 1, Group 1a, Group 2a, Group 3a, Group 1b, Group 2b, Group 3b, Group 1c, Group 2c, and Group 3c.

Table 6: Comparison of shear bond strength measurements by groups

	Group 1	Group 2	Group 3	Group 1a	Group 2a	Group 3a	Group 1b	Group 2b	Group 3b	Group 1c	Group 2c	Group 3c
Group 1	X	0.535	0.000*	0.008*	0.051	0.000*	0.183	0.048*	0.000*	0.113	0.854	0.000*
Group 2	X	X	0.000*	0.073	0.183	0.000*	0.550	0.135	0.000*	0.597	0.696	0.000*
Group 3	X	X	X	0.000*	0.000*	0.048*	0.000*	0.000*	0.679	0.000*	0.000*	0.066
Group 1a	X	X	X	X	0.581	0.000*	0.241	0.927	0.000*	0.103	0.024*	0.000*
Group 2a	X	X	X	X	X	0.000*	0.566	0.476	0.000*	0.190	0.118	0.000*
Group 3a	X	X	X	X	X	X	0.000*	0.000*	0.043*	0.000*	0.000*	0.383
Group 1b	X	X	X	X	X	X	X	0.370	0.000*	0.629	0.301	0.000*
Group 2b	X	X	X	X	X	X	X	X	0.000*	0.073	0.081	0.000*
Group 3b	X	X	X	X	X	X	X	X	X	0.000*	0.000*	0.198
Group 1c	X	X	X	X	X	X	X	X	X	X	0.370	0.000*
Group 2c	X	X	X	X	X	X	X	X	X	X	X	0.000*
Group 3c	X	X	X	X	X	X	X	X	X	X	X	X

*P<0.05 significant difference, P>0.05 no significant difference; Mann Whitney

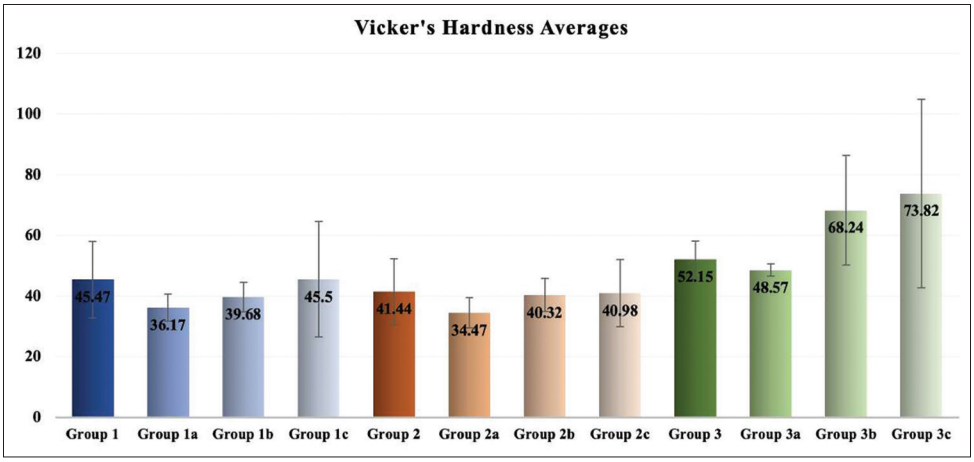

Figure 1: Distribution of Vicker's hardness measurement averages by groups

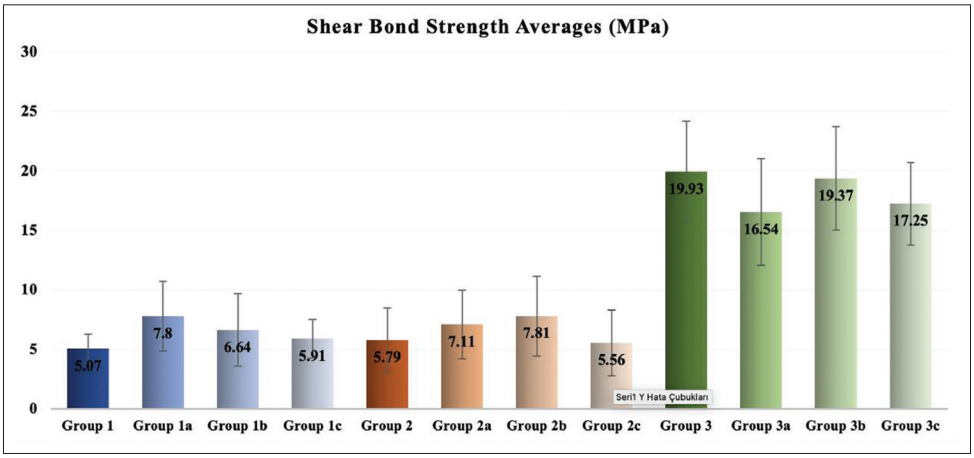

Figure 2: Distribution of averages of shear bond strength values by groups (MPa)

Discussion

There are few studies in the literature comparing the shear bond strength of high-viscosity GIC and FBF composite resin restorative materials. To our knowledge, no study has yet evaluated the effects of different high-viscosity glass ionomer and FBF composite resin restorative materials applied using different techniques (preheating application, ultrasonic activation application, and both at once) on the shear bond strength to dentin. Therefore, in this study, the shear bond

strength of reinforced high-viscosity GIC, high-viscosity GIC, and FBF composite resin to dentin was evaluated.

Lopes *et al*.[17] evaluated the surface microhardness of a preheated reinforced glass ionomer restorative material and noted that preheating increased the surface micro hardness of the material. This difference from our findings can be attributed to the fact that different amounts of heat were applied to the materials at different periods in the current study. In the aforementioned study, heat at a temperature of 54°C was applied to the reinforced high-viscosity GIC for 30 s. It can be thought that if the material is heated for a longer time, it will enter into an early hardening reaction, which may affect the material structure and harm the surface microhardness.

Although heat application has different effects on the stabilization time of the chemical bonds in GICs, it is believed that this technique increases the diffusion rate of ions and accelerates the hardening reaction, resulting in a reduction in the working and reaction times.[17] In the study by Kuter *et al*.,[18] the effect of heat application on the mechanical properties of GICs was evaluated, and it was reported that this technique increased the surface microhardness of the materials. This difference from our findings can be explained by the fact that in our investigation, less heat was applied to the materials before they were mixed in the amalgamator. In the aforementioned study, 80°C heat was applied after the materials were mixed and placed. However, in clinical practice, it does not seem possible to apply this amount of heat after glass ionomer restoration is completed.

The application of heat to GICs changes the molecular kinetic energy, promoting a more stable ionic exchange region; increasing the particle size of the materials; and providing a higher powder/liquid ratio owing to the removal of water that cannot provide a tight junction in the materials. In the study by Malul *et al*.,[19] heat treatment was applied to a reinforced high-viscosity GIC using different techniques, and the surface microhardness of the material was compared. It was noted that heat application improved the surface microhardness of the material. This difference from our findings can be attributed to the application of heat after mixing and placement of the material. External heat application after the curing reaction of materials is completed can positively affect the mechanical properties of materials.

In the study by Gorseta *et al*.,[20] the effect of preheating and ultrasonic activation of different GICs on the shear bond strength to enamel was evaluated, and it was reported that heat treatment improved the shear bond strength of the GICs to enamel. The researchers stated that changes in molecular kinetic energy due to high temperature caused the reorganization of molecules in the materials during curing; this molecular rearrangement could provide better bonding of the materials or obtain a more stable ionic exchange region despite a small temperature increase on the surface of the restoration. In another study by Gorseta *et al*.,[21] the effect of heating and ultrasonic activation of GICs on microleakage was evaluated, and it was noted that both techniques decreased microleakage by increasing the marginal adaptation of the materials.

Fagundes *et al*.[22] reported that ultrasonic activation increased the tensile bond strength of a GIC to dentin. This result supports that of current study. Some mechanisms are suggested to explain the effects of ultrasonic activation on the bonding of GICs to the dentin surface. 1) An increase in temperature can increase the rate of the reaction and the powder/liquid ratio owing to the evaporation of the liquid. 2) The mixing of particles and polyalkenoic acid chains by the waves produced during the movement of the tip may allow more frequent contact between the glass particles and acid. 3) Further, the high-frequency vibration of the material can reduce the volume and number of voids in the cement, allowing for more convenient and efficient placement and resulting in a denser material structure.

A high bond strength to dental tissues is an important factor in reducing the microleakage of restorative materials. As the bond strength increases, the marginal adaptation increases, thereby reducing microleakage. In the current study, preheating the reinforced high-viscosity GIC increased the shear bond strength to dentin, consistent with the findings of the two aforementioned studies.

Some of the advantages of applying the preheating technique to resin-containing materials include increasing the degree of conversion, improving the marginal adaptation of restorations by decreasing viscosity,[23-25] and reducing polymerization shrinkage. This technique also increases the conversion rate of monomers to polymers and the surface microhardness without accelerating the time at which maximum polymerization is achieved. This improvement is probably attributed to the increased molecular mobility and collision frequency of reactive molecules.[5] In the study by Lucey *et al*.,[26] the effect of preheating on the surface microhardness of a composite resin material was evaluated, and it was noted that 60°C heat application increased the surface microhardness of the material. The reason why these results are different from our results may be the use of a retractable composite resin in the aforementioned study as well as the difference in the amount of applied heat. The filler content, filler size, and monomer relationship with fillers are directly related

to the physical and mechanical properties of composite resins.[27] The success of the preheating technique depends on parameters such as material formulation, organic matrix type, inorganic filler ratio, applied heat, and heating time. In the current study, heat was applied to the FBF composite resin material. This technique may have had a negative effect on the material's structural properties by further increasing the flowability of the material, which had a flowable consistency. The surface microhardness of the material may have decreased owing to a negative effect on its structure.[5]

In this study, the highest shear bond strength was noted in the FBF composite resin restorative material. This value was found to be significantly higher than the shear bond strength of the reinforced high-viscosity GIC and high-viscosity GIC restorative materials in all test groups. The shear bond strength was found to be the highest in the control group, wherein the FBF composite resin material was applied by the manufacturer's instructions. Although the shear bond strength between the group in which the FBF composite resin was applied with ultrasonic activation and the group in which both heating and ultrasonic activation were applied did not significantly differ, these groups showed lower values than did the control group. However, the shear bond strength in the group in which the FBF composite resin material was placed with heating was found to be significantly lower than that in the control group. Increasing the temperature of composite resin restorative materials has been shown to increase the monomer mobility and polymerization reaction.[28] Silikas *et al*.[29] reported that the higher the degree of transformation in composite resin restorative materials, the higher the polymerization shrinkage. Polymerization shrinkage causes stresses between the tooth and restoration, failures of restoration to tooth bonding, and defects in the composite resin–tooth connection.[30] Accordingly, it is stated that the application of heat during the placement of FBF composite resin materials causes an increase in polymerization shrinkage due to an increase in the degree of monomer conversion of the materials and thus a decrease in the shear bond strength to dentin.

In the study by Kim *et al*.,[31] heating and sonic activation were applied to a flowable composite resin material, and the effect of these techniques on the penetration of the composite resin into pits and fissures was evaluated. The authors reported that heating and sonic activation increased the penetration of the flowable composite resin material into pits and fissures. Sonic activation decreased the viscosity of the material and yielded better penetration into pits and fissures. Another factor affecting the viscosity of the restorative material was temperature. The increase in temperature decreased the viscosity of the material, which affected the fluidity. In general, FBF composite resin materials contain a lower amount of filler than conventional flowable composite resin materials. This increased amount of filler aims to increase the depth of polymerization.[32] The difference in our results can be explained by the fact that the FBF composite resin used in this study and the traditional flowable composite resin material used in the aforementioned study have different chemical structures.

Khan *et al*.[33] reported that after 60 s of ultrasonic activation of two different composite resin restorative materials, the temperatures of the materials increased to 45°C and 46°C, respectively. With the increase in temperature, the mobility of free radicals in the materials increased, intensifying the polymerization reaction. Increasing the degree of conversion of monomers to polymers can also increase polymerization shrinkage-related stresses and adversely affect the bonding of composite resin materials to the prepared tooth surface.[34] In current study, 5 s ultrasonic activation was applied to the FBF composite resin, which did not affect the shear bond strength of the material to dentin. This finding can be explained by the fact that there was no significant change in the temperature of the material owing to the short ultrasonic activation time and that the technique did not cause a significant difference in the degree of monomer conversion.

In the study by Covey *et al*.,[7] the effect of heat application on the radial tensile strength of composite resin materials was evaluated, and it was noted that this technique had a positive effect on the diametral tensile strength. The researchers stated that heat treatment increased the mobility of both polymer segments and reactive free radicals formed during polymerization, consequently increasing the degree of conversion of monomers to polymers and allowing the crosslinking of polymers to increase. Most polymerization reactions occur within the first few minutes after light application. The polymerization reaction that continues after light application, also known as post-cure polymerization, can be influenced by factors such as the initial conversion degree of materials, composition of resins, presence of free radicals, and temperature of materials. Even after light treatment, reactive monomers remain present in the materials. However, the polymerization reaction is significantly limited owing to the reduced mobility of monomers. Increasing the temperature increases the mobility of monomers and the polymerization reaction. Taken together, increases in temperature allow the polymerization reaction to continue at a higher rate and for a longer time.[28] In the aforementioned study,

after the test specimens from composite resin materials were prepared, they were kept in an oven at 120°C for 7 min. The reason for the difference from the results of the current study may be that both the amount of heat applied and the duration of application differ, and the heat was applied after the materials had polymerized.

Davari *et al*.[35] evaluated the effect of different degrees of preheating on the microtensile bond strength of composite resin materials to the dentin surface. The authors noted that preheating at 37°C increased the microtensile bond strength of the tactile composite resin materials to dentin. Further, increasing the temperature decreased the viscosity of the materials, resulting in a higher tensile bond strength. Conversely, preheating the composite resin materials at 54°C significantly increased the monomer conversion compared with keeping the composite resin materials at room temperature. However, the higher the degree of transformation in the composite resin materials, the higher the polymerization shrinkage. Polymerization shrinkage combined with thermal shrinkage generated high interfacial tensions in the preheated composite resin restorative materials with adverse effects on marginal adaptation, integrity, and sealing.[34] The difference between the aforementioned study and this study can be attributed to the fact that the higher degree of preheating applied to the FBF composite resin material used in this study than in the aforementioned study damaged the structure of the material and hurt the shear bond strength to dentin.

Conclusions

Within the limitations of this *in vitro* study, the following conclusions can be made.

- Both preheating and ultrasonic activation of FBF composite resin materials can be recommended, as they increase the microhardness without adversely affecting the shear bond strength of the materials to dentin.
- Preheating reinforced HVGIC materials may be recommended, as it increases the shear bond strength to dentin.
- For HVGIC materials, there is no need for additional procedures since there is no advantage of heating and/or ultrasonic activation; the materials should be applied per the manufacturer's instructions.
- Additional treatments should not be recommended to shorten the clinical duration if there is no advantage in surface hardness or shear bond strength, as they will only increase the clinical application time.

Ethics statement

Ethics committee approval was received for this study by the Gazi University Faculty of Dentistry Clinical Research Ethics Committee with decision number "GÜDHKAEK.2021.05/2" dated 11.03.2021.

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Conflicts of interest

There are no conflicts of interest.

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