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Original Article

The Effect of Various Surface Treatments and Materials on the Bond Strength in the Repair of Glass Hybrid Restorative Material

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ABSTRACT

Objective: The purpose of this in vitro study is to evaluate the effect of various surface treatments (STs) and restorative materials on the shear bond strength (SBS) of glass hybrid (GH) restorative materials.

Methods: Sixty-three GH restorative material samples (Equia Forte Fil, GC) were prepared (6 × 2 mm) and randomly divided into 3 groups according to the subsequent ST (n = 21): phosphoric acid etching (PA), aluminum oxide sandblasting (AO), and diamond bur roughening (DB). After that, 3 subgroups were created from the samples in each group based on the restoration material applied: Ionofil Molar AC (IM; Voco), Fuji II LC (FLC; GC), and Charisma Smart (CS; Kulzer). The bonded specimens were subjected to the SBS test. In addition, the samples were evaluated with a scanning electron microscopy. The results were statistically examined using two-way analysis of variance with post hoc test ($P = .05$).

Results: The CS groups were higher in SBS compared to the other groups, whereas the IM groups had low bond strength ($P < .05$). The FLC, CS, DB, and PA groups showed the highest SBS ($P < .05$). The lowest SBS values were observed in the PA group in IM ($P < .05$).

Conclusion: The GH restorative materials were successfully repaired with composite resin using diamond bur roughening or phosphoric acid etching.

Keywords: Bond strength, composite resin, glass hybrid restorative material, repair

INTRODUCTION

Nowadays, the goal of contemporary dentistry is to treat demineralized dentin using adhesive restorative materials while removing as little tooth tissue as possible.[1] For this purpose, composite resins have been used for years in accordance with the minimally invasive treatment procedure and high adhesive bonding potential. The rapid development of restorative treatment methods in connection with materials and application techniques have made the clinical use of glass ionomer cements (GIC) quite widespread. Glass ionomer cements have unique properties including adherence to moistened dental structures, low-toxicity, anticariogenic effects because of fluoride release, thermal compliance with tooth hard tissues, and biocompatibility.[2] Despite these advantages, low mechanical properties, poor aesthetics, and a tendency to degradation in acidic environments have limited the use of GICs as permanent restoration materials, especially in load-bearing areas.[3,4] To eliminate the disadvantages of glass ionomer cements, new GIC forms have been presented on the market today. One of them, encapsulated conventional GICs, can be used in high stress bearing areas such as class I cavities and is claimed by manufacturers to have high wear resistance.[5] Also, with the new generation glass hybrid (GH) restorative materials developed as permanent restorative materials, the inadequate mechanical properties of conventional GICs and their abrasion resistance to strong occlusal stresses have improved, and the fields of use limited to class I and class V cavities as restorative materials have expanded. The manufacturer claims that the matrix of

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this new system combines fillers, fluoroaluminosilicate glasses of different sizes similar to hybrid composite resins.[6] In addition, they are now frequently preferred by clinicians due to their fluoride release and biocompatibility with tooth tissues.[7]

One of the major problems encountered in the clinic is restoration failure. High mechanical strains, pH fluctuations, salivary enzymes, thermal changes, and fractures can all cause permanent restorations to deteriorate.[8] Clinicians often replace entirely damaged restorations with new ones or partially remove old restorations and restore them with new ones. Excessive tooth structure could be lost if unsuccessful restorations are completely replaced, inevitably leading to enlargement of the preparation and may lead to damage the pulpal tissue.[9] For these reasons, it is becoming increasingly important to remove only the defective part of the restoration and repair it by placing a biomaterial there.[10] Successful repair is affected by the surface treatments (STs) to be applied as well as old and new restorative materials. With the application of STs, the repaired surface area and surface energy are increased, while the layer that has changed on the surface is removed.[11] For this purpose, micro- or macro-mechanical and chemical surface preparation protocols such as phosphoric or hydrofluoric acid etching, aluminum oxide (Al_2O_3) particles sandblasting, diamond bur roughening, and using laser beams and bonding agents can be applied.[12,13] Restorative materials have different distributions, morphologies, sizes, and chemical compositions of filler, therefore they may respond differently to various repair techniques.[14]

Even though various ST protocols and materials on bond strength for repair of different restorative materials have been widely studied, there is limited research investigating the repair of GH restorative material in the literature. Thus, the goal of this study was to determine the various STs and restorative materials on bond strength of GH restorative materials. The null hypothesis of the study was that no difference would be observed for bond strength of various ST protocols and repair restorative materials used for the repair procedure of the GH restorative materials.

MAIN POINTS

- The most important finding of the study is that the glass hybrid (GH) restorative material could be repaired at clinically acceptable bond strength values.
- The successful repair of GH restorative material with composite resins provides benefits in terms of the indication of resin restorations, which are increasingly used nowadays.
- The clinical ease of use of diamond burs and phosphoric acid would help repair procedures result in shorter times and increased patient satisfaction.

METHODS

The materials used in this study are shown in Table 1. For the preparation of the GH restorative material samples to be repaired, 63 plastic molds with the dimensions of 15 × 10 mm, with a cylindrical cavity of 6 × 2 mm on 1 surface, were fabricated. The GH restorative material (Equia Forte Fil, Shade A2, GC, Japan) was placed in plastic molds following the manufacturer's instructions and a cellulose acetate strip as well as 1 mm thick cement glass were placed on it. To obtain a smooth surface and overflow of excess material, slight pressure was applied. When the GH had time to cure, removal of the glass slab and transparent matrix tape was done followed by excess material removal with a 1200-grit silicon carbide paper. The specimens were slightly dried and the coating substance (Equia Forte Coat, GC, Japan) was applied on the upper surface and subjected to 20 seconds of light cure per side using a 1000 mW/cm^2 LED polymerization device (Valo Cordless, Ultradent, South Jordan, UT, USA). The specimens were stored in 37°C distilled water for 24 hours and then randomly divided into 3 groups and based on the following STs ($n=21$):

I. (PA): The GH restorative material surfaces were etched with 37% phosphoric acid (Scotchbond Etchant; 3M ESPE, St. Paul, Minn, USA) for 30 seconds, followed by rinsing and drying with air.

II. (AO): The GH restorative materials were sandblasted with a sandblaster (AirsonicVR, Duisburg, Germany) for 10 seconds using Al_2O_3 (Cobra; 50 mm Al_2O_3, Renfert GmbH, Hilzingen, Germany) at a 10 mm distance from the resin and 2.5 bar pressure. After processing, the samples were rinsed and dried with air.

III. (DB): The GH restorative material surfaces were roughened with a diamond coarse fissure bur (DIATECH, Swiss Dental, Heerbrugg, Switzerland) under water cooling. The samples were rinsed after processing and dried using air.

After the STs, each group of samples was separated into 3 subgroups according to the applied repair material, and the repair materials were prepared by placing a cylindrical plastic tube (3 × 4 mm) over the surface-treated GH restorative material ($n=7$): Ionofil Molar AC (IM; Voco Cuxhaven, Germany), Fuji II LC (FLC; GC Corp, Japan), or Charisma Smart (CS; Kulzer GmbH, Hainau, Germany). All the repair materials were applied according to the instructions of the manufacturer, and a color A2 was used. The Single Bond Universal adhesive (3M ESPE, St. Paul, Minn, USA) was applied to the surfaces of the GH restorative material surfaces in CS groups before the repair material

Table 1. Materials Used in the Study

Product name	Type	Manufacturer	Composition
Equia Forte Fil	Glass hybrid restorative material	GC, Tokyo, Japan	Fluoroaluminosilicate glass, polybasic carboxylic acid, polyacrylic acid, distilled water
Equia Forte Coat		GC Corporation, Tokyo, Japan	25%-50% methyl methacryl, 10%-15% silicon dioxide, 0.09% camphoroquinone, 30%-40% urethane methacrylate, 1%-5% phosphoric ester monomer
Ionofil Molar AC	Conventional glass ionomer cement	Voco/Germany	Polyacrylic acid, tartaric acid, aluminofluorosilicate glass
Fuji II LC	Resin modified glass ionomer cement	GC, Tokyo, Japan	Aluminofluorosilicate glass, Poly-HEMA
Charisma Smart	Submicron-hybrid composite	Kulzer GmbH, Hanau, Germany	Resin matrix: Bis-GMA, Bis EMA, TEGDMA Filler: 0.5-10 µm particles; 20 nm particles, glass ceramic fillers, functionalized SiO_2 weight 89%–volume 73%
Single Bond Universal	Universal dental adhesive	3M ESPE, St Paul, Minn, USA	10-MDP phosphate monomers, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, fillers, ethanol, water, initiators, silane

10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate.

was applied and cured with an LED curing light. After finishing this process, all the samples were stored in a 37°C water for 24 hours.

Shear Bond Strength Test

The shear bond strength (SBS) test was accomplished with a universal test machine (Lloyd LRX, Lloyd Instruments, Farnham, USA) at a crosshead speed of 1 mm/min. A force was applied straight to the repair site until breakage occurred, using an end-flattened steel rod. SBS was given in units of MPa and calculated by dividing the max load (N) at fracture by the adhesion area (mm^2).

Fractured areas were observed using a stereomicroscope at 25× magnification to help decide the fracture mode of samples. The type of failure was determined to be adhesive failure, cohesive failure, or mixed.

Scanning Electron Microscopy Evaluation

One sample randomly selected was prepared for the scanning electron microscopy (SEM) evaluations for the treatment protocol of each surface and repair restorative materials. The samples were treated with gold using sputtering, and investigated by SEM (FEI Quanta 450 FEG, Hillsboro, OR, USA). The SEM images obtained were from the upper surface that had been surface treated and repaired GH restorative materials at 100× and 500× magnifications.

Statistical Analysis

Statistical analysis was done using Statistical Package for the Social Sciences Statistics software, version 23.0 (IBM Corp.; Armonk, NY, USA). Data underwent a Shapiro-Wilk test to determine normality. A two-way analysis of variance (ANOVA) was done to compare values based on the repair material and ST. Multiple comparisons using Tukey HSD and Tamhane's T2 tests were performed. A *P*-value of less than .05 was considered statistically significant.

RESULTS

The average SBS for the tested groups is summarized in Table 2. The statistically significant difference between the groups was shown by two-way ANOVA. Irrespective of repair material, the ST protocols did not affect SBS values in any group. Though, the types of repair restorative materials had a substantial effect on bond strength. No interaction was found for these 2 variables ($P = .016$).

In a comparison between the repair restorative materials, a significant difference was determined for the groups ($P < .05$). Repair with the CS demonstrated a significant

Table 2. The Mean Shear Bond Strength Values (MPa) and Standard Deviations (±SD) of all Tested Groups

Surface Treatments	Repair Materials		
	IM	FLC	CS
PA	0.17 ± 0.07^{D}	6.47 ± 1.99^{BC}	11.16 ± 1.34^{A}
AO	2.22 ± 0.73^{E}	5.19 ± 1.81^{CE}	10.71 ± 2.07^{AB}
DB	0.30 ± 0.13^{D}	6.73 ± 2.07^{BC}	11.16 ± 1.05^{A}

There is no difference between interactions with the same letter.
AO, aluminum oxide sandblasting; CS, Charisma Smart; DB, diamond bur roughening; FLC, Fuji II LC; IM, Ionofil Molar AC; MPa, megapascal; PA, phosphoric acid etching.

bond strength compared to the repair with IM and FLC, while the lowest bond strength was observed in the IM (P < .05). In the CS and FLC, the PA and DB treatment groups demonstrated the highest bond strengths (P < .05). No statistical difference was found for STs (P > .05). The AO treatment group showed a statistically significant difference compared to the other STs in the IM group (P < .05). In the same group, no statistical difference was found for PA, which showed the lowest bond strength, and DB (P > .05).

Figure 1 shows modes of failure for the samples studied after the SBS tests. Fracture analysis was found to be consistent with the determined SBS. Excluding the IM group, the predominant failure was cohesive in restorative materials. The PA treatment group in CS had the highest cohesive failure rate. The adhesive failure mode in the restorative material was only observed in the IM group.

The SEM image of each of the ST protocols and repair restorative materials is shown in Figures 2, 3, and 4. The corrugated and irregular structure produced by the roughening process in the DB groups can be easily noticed. Shallow and unidirectional grooves were observed in AO treatment samples. It was observed that the surface of the CS showed a smoother appearance than the GH in the mixed fractures in the CS groups. The repair surfaces in the IM groups was visible and showed a predominantly smooth surface. Adhesive failure was identified as the failure mode (Figure 1).

DISCUSSION

Although GIC provides advantages such as chemical adhesion and anti-cariogenic qualities resulting from fluoride release, its use is constrained by its poor mechanical qualities. With current developments, the introduction of highly branched polyacrylic acid products containing nano-sized glass particles with high molecular weight has enabled the development of the properties of GICs and their use as permanent restorations.[15] GI restorative materials may need to be repaired or replaced more frequently than restorative materials like amalgam and composite resin since they are more brittle and less resistant to abrasion.[16]

In the repair of restorations, it is often not possible to determine which restorative material was used in the old restoration. Different restorative materials are generally used in such cases as repair, and different surface protocols are applied to increase the repair strength.[17] Although

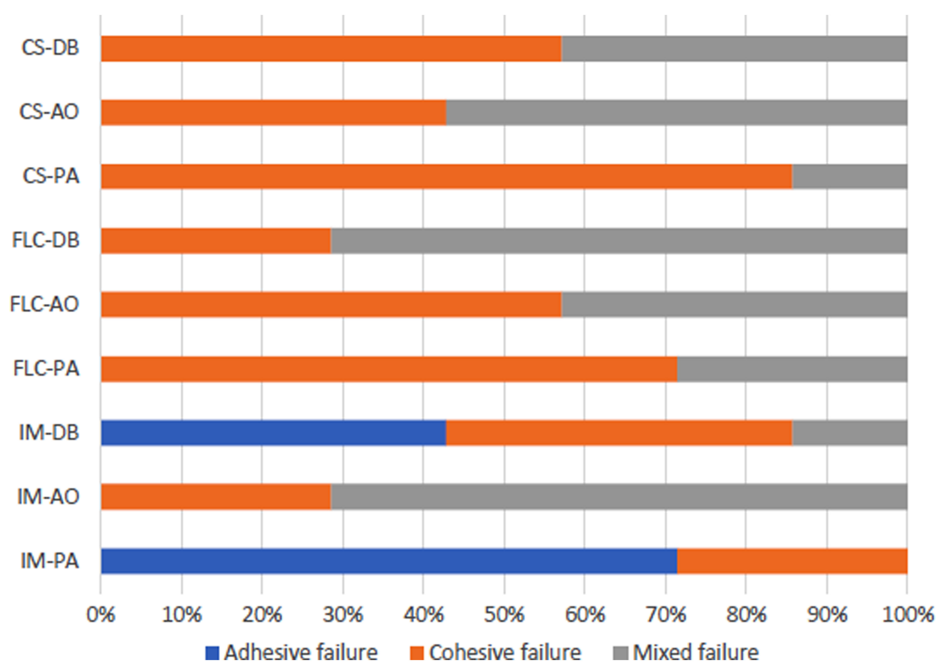

Figure 1. Graphic representation of the failure modes for each group (%).

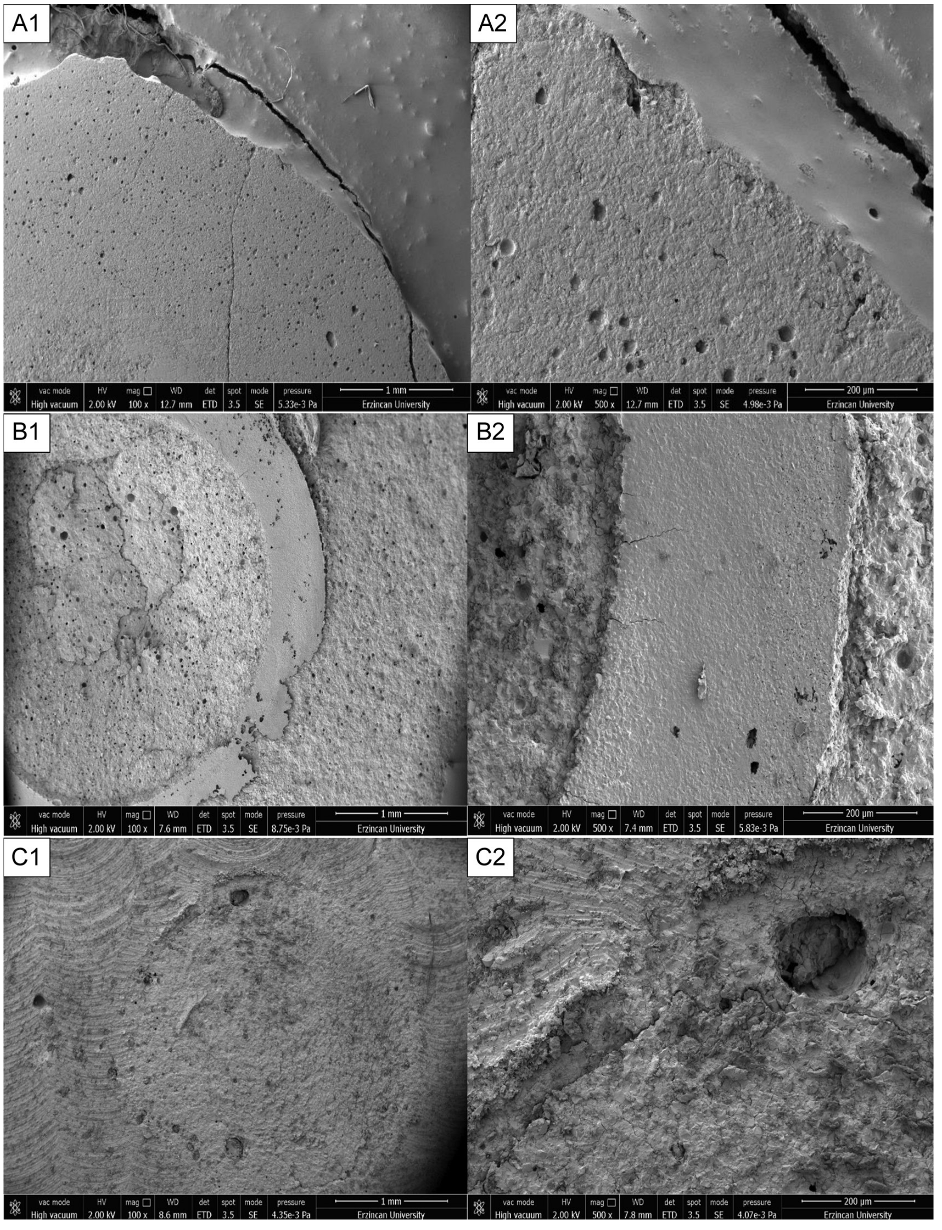

Figure 2. SEM images of fractured surface of three specimens from Ionofil Molar AC at (1) × 100 and (2) × 500 magnification: (A) phosphoric acid etching, cohesive failure; (B) Al_2O_3 sandblasting, mixed failure; (C) diamond bur roughening, adhesive failure.

there are many studies in the literature regarding the repair of resin modified glass ionomer and composite resin,[18-20] the number of studies on the GH restorative materials' repair potential are very few.[21,22] Therefore, this study aimed to determine the effects of different STs and restorative materials on SBS of GH restorative materials. The null hypothesis of no differences between bond strengths of different STs and restorative materials used for the repair procedure of the GH restorative material was rejected based on our findings.

Among the restorative materials tested, CS showed the highest values of bond strength over the FLC and IM materials for all the repair STs. Meral et al.[21] repaired Equia Forte Fil with 3 different restorative materials and reported that the highest bonding values were obtained in the composite resin groups. Vural et al.[22] investigated the repair of Equia Forte Fil with different STs and restorative materials and reported the highest values in the composite resin groups. In this study, Single Bond Universal was used after the ST in the CS repair groups. Single Bond Universal is a universal adhesive system with 10-methacryloyloxyde cyldihydrogen phosphate (10-MDP) content. 10-MDP is formed ionic bonds with calcium salts.[23] Calcium ions attached to the carboxyl groups of the polyacrylic acid chains in the structure of the GH restorative material can interact with the MDP on the cement surface and increase the strength of the bond.[21] Furthermore, silane found in

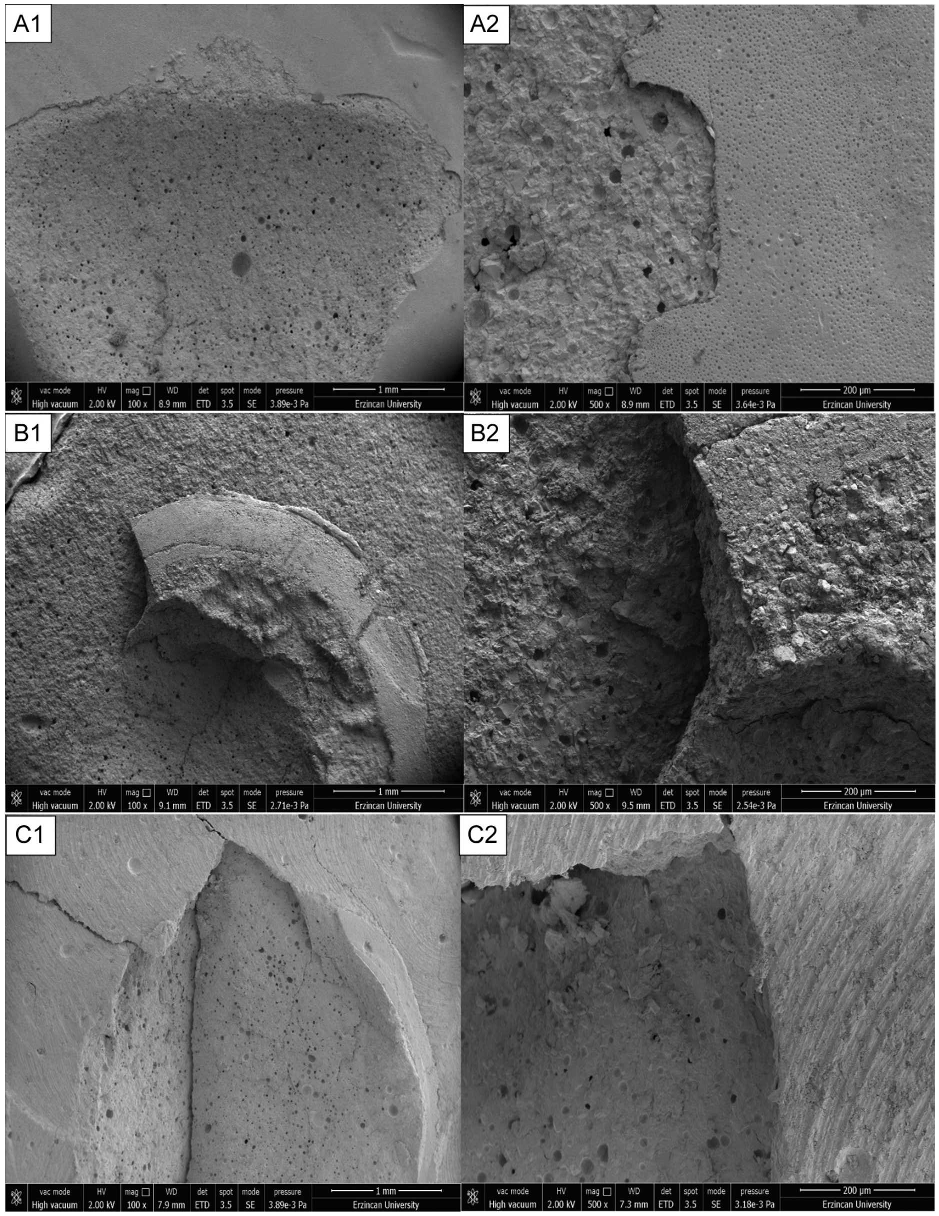

Figure 3. SEM images of fractured surface of three specimens from Fuji II LC at (1) ×100 and (2) ×500 magnification: (A) phosphoric acid etching, cohesive failure; (B) Al_2O_3 sandblasting, cohesive failure; (C) diamond bur roughening, mixed failure.

the Single Bond Universal may split off hydroxyl groups and connect to the surface through oxygen bridges. Due to their ability to bond with fillers and organic matrix, silanes are commonly used for bonding and repair. They also enhance surface wettability by modifying surface energy.[24] According to earlier studies, using a universal adhesive with silane in it or applying silane before adhesive processes increased the bond strength.[24,25] Based on this information, the high bond strength of the CS groups can be attributed to the increased wettability of the universal adhesive used with the irregularities created by the various repair procedures on the GH restorative materials. In the study, the FLC groups showed higher SBS compared to IM for all the ST procedures. Contrary to IM, the FLC groups' high values can be related to their resin composition including hydroxyethyl methacrylate (HEMA). HEMA is a low weight hydrophilic monomer that wets the substrate, and readily incorporated into adhesive formations.[26] HEMA in the FLC may have improved the adherence of materials to the surface.

The clinical repair process is dependent on the bonding capacity of the old restorative composition with the new repair material. Optimum adhesion is dependent on the surface characteristics and the properties of the chemical adhesive used, as well as the chemical properties and viscosity of the materials.[27] In the study, IM was found to be the restorative material with the lowest bond strength.

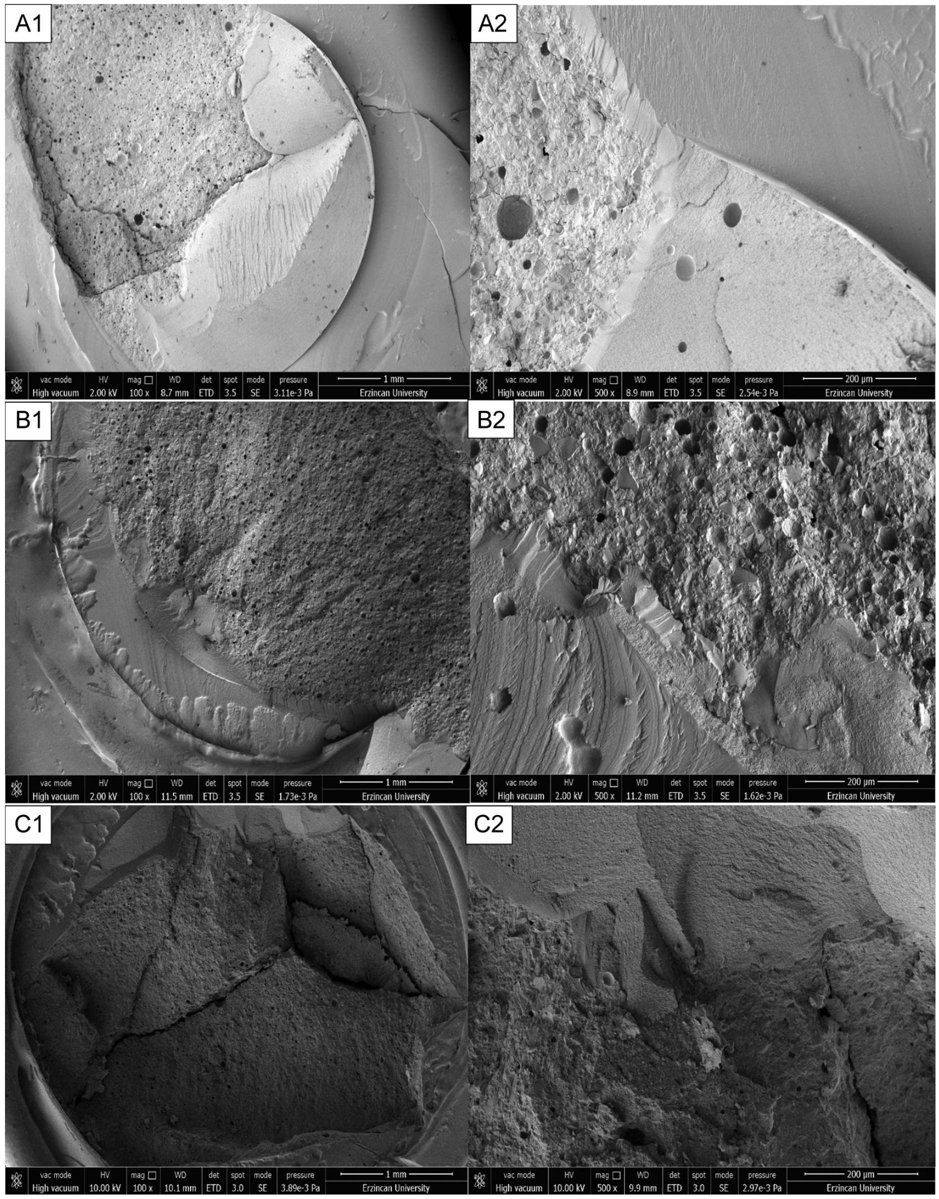

Figure 4. SEM images of fractured surface of three specimens from Charisma Smart at (1) × 100 and (2) × 500 magnification: (A) phosphoric acid etching, mixed failure; (B) Al_2O_3 sandblasting, cohesive failure; (C) diamond bur roughening, mixed failure.

IM is a conventional encapsulated glass ionomer cement. When the literature was investigated, no published studies could be found on the repair of GH restorative materials with conventional glass ionomer cement. The reason why the IM group shows the lowest bond strength compared to other repair materials in all repair processes may be that it does not flow sufficiently on rough surfaces due to its high viscosity and low wettability, which prevents optimum flow.

ST protocols are important for adhesion in the repair of restorative materials. Increased surface roughness is critical both for improving SBS between the new and old restoration and promoting mechanical interlocking. Even though many STs in repair processes are used, a consensus has yet to be reached on the most ideal. Due to the data about the effect of STs on GH restorative material repair, different STs were used in the study.

In the study, the bond strength with the highest values was the PA and DB treatment groups in CS and FLC, and no statistical difference was observed for the 2 ST groups. Vural et al.[22] stated that the highest repair µTBS between GH and composite resin was achieved when the GH was roughened with a diamond bur or etched with phosphoric acid. In addition, they stated that the highest repair µTBS between the GH and composite resin was formed with the universal adhesive following roughening with a diamond bur or etched with phosphoric acid. In this study, the lowest SBS was observed in AO groups in CS and FLC. There are studies in the literature that report no improvement on bond strength following sandblasting with Al_2O_3.[28,29] Da Costa et al.[30] reported that sandblasting could not roughen the material surface as much as a diamond bur in composite repair. In addition, there are conflicting results in the literature.[31,32] In a study investigating the effect of different repair procedures on the repair bond strength of composite resins, it was determined that no difference was observed for repair bond strength following sandblasting with Al_2O_3 and roughening with a bur.[32] The high bond strength of the PA and DB treatments in the CS and FLC groups in this study may be due to sufficient roughening together with the chemical structure of the repair materials that increase the bonding. The differences found in previous studies may be linked to the different protocols and materials used.

In this study, the bond strengths were lower in the PA and DB groups in the IM, while the AO treatment showed a statistically higher bond strength in comparison to other STs in the same group. Arslan et al.[33] stated that in the SEM examinations of their studies, high peak points and deep pits were formed on the material surface of the diamond bur roughening. Since sandblasting with Al_2O_3 is in microparticle sizes, it is thought that the surface is flatter, but the roughness distribution is denser. While the deep rough structure formed by the diamond bur supports the bond strength for FLC and CS, whose wettability is higher than IM; for the IM group, which prevents the ideal surface flow without any wetting agent, it may have caused an insufficient bond strength. In the AO group, the shallow and densely rough surface may have caused the IM group to show higher bonding values compared to other STs.

Kalra et al.[34]showed the lowest required bond strength value of a repair material to be applied in the mouth to be 8-9 MPa, considering the chewing forces. According to the results of this study, these values were achieved only in the CS group. However, it is stated that it should be taken into account that these values may be affected by the brands of the materials used, and studies with different brands may be beneficial.

The results in the study were obtained under in vitro conditions and real oral conditions such as chewing forces, wear, and fatigue cycling were not simulated. Therefore, long-term durability of repaired GH restorative material even with different types of restoratives and also with varying ST methods should be evaluated further.

In conclusion, within the limitations of this study, it was found that glass hybrid restorative materials can be repaired successfully. In the repair prognosis, an efficient and safe repair protocol is a critical factor. Based on the outcomes of the study, silane-containing adhesive combined with etching with phosphoric acid or roughening with a diamond bur can be suggested as a recommended procedure for the repair of glass hybrid restorative materials. The findings of this study may show a positive outcome that involves the repair of glass hybrid restorative materials as permanent restorative materials.

The dentist does not have the chance to determine the existing restoration material in the tooth in order to achieve success while performing the restoration repair. Success in repairing a restoration is only possible with the conscious selection of the surface preparation method, bonding system, and repair material. It is expected that the results of the study will shed light on dental practice in this respect. With the increase in minimally invasive applications such as repair in modern dentistry, we believe that the quality of "repairability" should be added to the sought-after properties of restoration materials. We expect that the results of this study, which was designed according to very different parameters, can contribute to this issue as well.

Ethics Committee Approval: This study was carried out in vitro in a laboratory environment. The methods and dental materials used have not been applied to any living creature. Therefore, an ethical committee was not required and no application was made in this direction.

Informed Consent: Informed consent was waived since this study was not carried on any living creature.

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Declaration of Interests: The authors declare that they have no competing interests.

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