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The Effect of Micromechanical Surface Preparation and Adhesive Surface Modification Strategies on Resin-Matrix Ceramic Repair Bond Strength Using Universal Adhesive Containing Silane Agent

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Abstract

Objectives This research assesses the effect of the different micromechanical surface preparations and chemically adhesive surface modification strategies applied to resin-matrix ceramics (Shofu Block HC) repaired using resin composites.

Materials and Methods Eighty resin-matrix ceramics were conducted and designed into eight groups of 10 specimens and surface treated with (1) micromechanical preparation with sandblast (SB) or hydrofluoric acid (HF), and (2) chemically adhesive techniques with HC primer (HC) and/or silane (Si) and/or conventional adhesive (AD) or universal adhesive (UA) designing are as follows: group 1, SB + HC; group 2, SB + HC + AD; group 3, SB + HC + Si + AD; group 4, SB + HC + UA; group 5, HF + HC; group 6, HF + HC + AD; group 7, HF + HC + Si + AD; and group 8, HF + HC + UA. An ultradent model was put on the specimen center, then filled resin composite. Mechanical testing instrument was used to determine the samples' microshear bond strength (MSBS). To inspect failure modes, a stereomicroscope was used for observing the debonded surfaces.

Statistical Analysis To assess the data, a one-way analysis of variance was employed, and the significant level ($p < 0.05$) was established with Tukey's test.

Results Group 3 (29.29 ± 2.58 MPa) and group 4 (28.34 ± 1.26 MPa) demonstrated the two maximum MSBS values. The minimum MSBS (10.02 ± 3.31 MPa) was discovered by group 5. Nevertheless, group 2's MSBS values (22.78 ± 2.44 MPa) differed significantly from the values for groups 3 and 4. All the fractured samples in groups 1, 5, 6, 7, and 8 had an adhesive failure pattern. Furthermore, group 3 presented the greatest mixed failures (40%).

Conclusion The SB is the most effective protocol for producing micromechanical retention. The application of HC primer and Si agent prior to the adhesive agent is the best chemical adhesive strategy for sandblasted resin-matrix ceramic surfaces. Additionally, the application of HC primer before the use of UA containing acid-resistant Si is the best alternative chemical adhesive strategy for improving the MSBS.

Keywords
- adhesive agent
- hybrid ceramic
- repair bond strength
- surface treatment

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Introduction

Over the past few years, dental computer-aided design/ computer-aided manufacturing (CAD/CAM) technologies have advanced quickly and are now an essential aspect of restorative dentistry and prosthodontics. A variety of restorative materials are already accessible as a result of CAD/CAM technology, including resin-matrix ceramics, polycrystalline ceramics, and glass-matrix ceramics.[1] Recently, resin-matrix ceramics have launched CAD/CAM materials that include ceramics with color stability and strengthening mechanisms, as well as those that combine the features of polymers with minimal opposing wear and improved flexural strengths.[2,3] Resin-matrix ceramics are easier to mill and repair,[4] have low abrasiveness, and polish easily for glossy, smooth surfaces.[5] However, the drawbacks of glass-matrix ceramics remain the necessity for postfiring, difficulties in machinability, and brittleness.[1,6]

Although improvements in resin-matrix ceramic CAD/ CAM materials have been made, fractures are still possible due to a wide variety of factors.[7] Restoration repair or restoration replacement are the two options available for handling fractured restorations.[8] In contrast to restoration replacement, intraoral repair is a procedure that is achievable, affordable, and conservative. The reliability and durability of the bond that links the broken restoration with the repair substance, such as resin composites, are crucial for the effective implementation of an intraoral repair strategy.[4,9] Micromechanical surface preparation and chemically adhesive surface modification techniques are used in restoration repair to provide an effective bond between the resin-matrix ceramic and the resin composite.[4,10] Hydrofluoric acid (HF) etching and pressure airflow abrasion are two examples of micromechanical surface preparation techniques.[4] For chemically adhesive surface modifications, the use of adhesive approaches, including silane (Si) coupling agent, conventional adhesive (AD), or universal adhesive (UA), is recommended to strengthen the repair bond ability after micromechanical surface preparation.[4,11] However, Si-free conventional AD substances need multiple processes and technical sensitivity, whereas innovative UAs that integrate Si ease the clinical approach and minimize the number of application processes.[12,13]

There are limited investigations exploring the effect of micromechanical surface preparation and chemically adhesive surface modification strategies on resin-matrix ceramic CAD/CAM block repair bond strength using resin composites.[4,11,14] The intention of the current investigation was to investigate the different micromechanical surface preparations and chemically adhesive surface modification strategies applied to resin-matrix ceramics (Shofu Block HC; Shofu, Kyoto, Japan) that were repaired using resin composites, especially using conventional ADs and UAs containing Si coupling agents. It was the null hypothesis that resin-matrix ceramics (Shofu Block HC) repaired using resin composites would not differ in micromechanical surface treatments and chemically adhesive strategies across protocols.

Materials and Methods

Resin-Matrix Ceramic Preparation

The resin-matrix ceramic CAD/CAM materials, mainly Shofu Block HC (Shofu), were investigated in this work. An Accuton-50 wafer cutting equipment (Struers, Ohio, United States) measuring 6 × 7 mm and 1.5 mm thick was used to cut the 80 pieces into a rectangular shape. The thermocycling machine (Proto-tech, Microforce, Oregon, United States) was used to age the resin-matrix ceramic specimens. It cycled the material 5,000 times between 5°C and 55°C, allowing 30 seconds for dwell time and 5 seconds for transfer.[4] The samples were put in a polyvinyl chloride tube that was filled with epoxy resin. The specimen surfaces were resurfaced using 600-grit silicon carbide on a 3M abrasive sheet (3M, Minnesota, United States) to uniformly adjust the surface roughness. Using ultrasonic cleaning, all of the samples were submerged in distilled water for approximately 10 minutes.

►**Table 1** provides a brief description of the materials utilized in this investigation.

Sandblast Protocol

The aged resin-matrix ceramic specimens were subjected to a 10-second exposure to 50-micron Al_2O_3 particles spaced 10 mm apart under two bars of pressure.[4] The specimens underwent sandblasting, cleaning, and 10 seconds of air drying using a triple syringe.

Hydrofluoric Acid Etching Protocol

The aged resin-matrix ceramic specimens were treated with 9% HF (Ultradent Products, South Jordan) for 1 minute,[15] and then were cleaned and allowed to air dry using an oil-free air/water syringe.

HC Primer Application Protocol

The HC primer (Shofu) was primed to the specimen's surface using a microbrush for 20 seconds. The surplus primer was then removed using a fresh microbrush and left to air dry for another 20 seconds. Then, applying a light-emitting diode curing apparatus (Demi plus, Kerr Corporation, California, United States), the device was light activated ~10 seconds in compliance with the instructions provided by the manufacturer. It was not light activated if the HC primer was treated prior to the application of the UA.

Silane Coupling Agent Application Protocol

The specimen's surface was treated with the Si coupling agent (RelyX ceramic primer, 3M) using a microbrush for a duration of 1 minute.[16] After letting the Si coupling agent air dry for around 10 seconds, the solvent was carefully removed.

Conventional Adhesive and Universal Adhesive Application Protocol

The adhesive was coated to the specimen's surface for ~20 seconds with a microbrush. A fresh microbrush was applied to remove any leftover adhesive agents. The adhesive's solvent was eliminated by letting it air dry for approximately 5 seconds. It was permitted to air blow it dry until the liquid

Table 1 Resin materials that were utilized for this research

Materials	Compositions
Shofu Block HC (Shofu, Kyoto, Japan); Lot: 0721594	UDMA, TEGDMA, filler; silica powder, microfumed silica, zirconium silicate, 61% by weight
HC primer (Shofu, Kyoto, Japan); Lot: 102210	10–20% MMA, 10–20% acetone, UDMA, polymerization initiator, and others
Adper single bond 2 (3M ESPE dental products, Minnesota, United States); Lot: 9910162	Ethanol, water, Bis-GMA, HEMA, UDMA, EDMAB, silane treated silica, glycerol 1,3 dimethacrylate, copolymer of acrylic and itaconic acids, diphenyliodonium hexafluorophosphate
RelyX Ceramic primer (3M, Minnesota, United States); Lot: N988623	Ethanol, water, 3-MPTS
Beautibond Xtreme (Shofu, Kyoto, Japan); Lot: 042343	Phosphate ester, dithiooctanoate and carboxylic acid monomers, acid-resistant silane coupling agent, acetone
Resin composite (Harmonize A3E shade, Kerr Corporation, California, United States); Lot: 9127134	Bis-GMA, TEGDMA, EBPADMA, zirconia/silica cluster filler (2–3 μm) comprised 20 nm spherical fumed silica and 5 nm zirconia particles, prepolymerized filler

Abbreviations: 3-MPTS, 3-methacryloxypropyltrimethoxysilane; Bis-GMA, bisphenol A-glycidyl methacrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; HEMA, 2-hydroxyethyl methacrylate; MMA, methylmethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

stopped moving and the surface became glossy. It was then given a 20-second light activation.

Resin Composite Application Protocol

Random assignments were made to eight groups ($n = 10$ per group) based on the resin-matrix ceramic surface-treated specimens. (1) Micromechanical surface preparation with sandblast (SB) or HF and (2) chemically adhesive surface modification techniques with HC primer (HC) and/or Si coupling agent and/or conventional AD (Adper single bond 2, 3M ESPE dental products, Minnesota, United States) or UA (Beautibond Xtreme, Shofu) were used as follows:

Group 1: SB + HC.
Group 2: SB + HC + AD.
Group 3: SB + HC + Si + AD.
Group 4: SB + HC + UA.
Group 5: HF + HC.
Group 6: HF + HC + AD.
Group 7: HF + HC + Si + AD.
Group 8: HF + HC + UA.

An ultradent model with a thickness of 2.0 mm and a diameter of 2.0 mm was positioned in the middle of the surface-treated specimen. The nanofiller resin composite (Harmonize, Kerr Corporation) was inserted into the ultradent model and then light activated for ~40 seconds. After removing an ultradent model, light activation was repeated for 40 seconds. Every sample was incubated for a full day at 37° C in a laboratory incubator chamber (Contherm Scientific Ltd., Lower Hutt, New Zealand) that contained distilled water.

Microshear Bond Strength and Failure Pattern Examination

The microshear bond strength (MSBS) values were assessed utilizing a universal measurement tool with a knife-edge blade at an experimental speed of 0.5 mm per minute (AGS-X 500N, Shimadzu Corporation, Kyoto, Japan). The MSBS value was calculated by dividing the area of the adhesion zone by the bond breakdown strength.

The CAD/CAM material block and resin composites' fracture mode patterns were observed under a stereomicroscope with a ×50 magnification. Three patterns were created to identify the fracture modes: (1) an adhesive pattern (fracture on the connection between CAD/CAM block and resin composite), (2) a cohesive pattern (fracture inside CAD/CAM block or resin composite), and (3) a mixed pattern (when taken together, cohesive and adhesive failure patterns).

The Data's Statistical Analysis

A one-way analysis of variance test was employed to analyze the data to ascertain how micromechanical surface preparation and chemically adhesive surface modification strategies affected MSBS. For pairwise comparison, the Tukey's honestly significant difference test was used. Version 20.0 of IBM SPSS was employed for all statistical analyses ($p < 0.05$).

Results

In ►**Table 2**, the mean MSBS values and standard deviation are provided. Group 3 (29.29 ± 2.58 MPa) and group 4 (28.34 ± 1.26 MPa) demonstrated the two maximum MSBS values. The significantly minimum MSBS value (10.02 ± 3.31 MPa) was discovered by group 5. In comparison to group 1 (16.12 ± 1.54 MPa), the bond strength values of group 6 (14.98 ± 1.56 MPa), group 7 (15.48 ± 2.15 MPa), and group 8 (15.02 ± 1.64 MPa) did not differ significantly. Nevertheless, MSBS values of group 2 (22.78 ± 2.44 MPa) differed significantly from the values for groups 3 and 4.

A brief summary of the failure-type incidence pattern appears in ►**Table 2**. All of the fractured samples in groups 1,

Table 2 The mean MSBS ± SD and percentage of failure pattern

Groups	Mean MSBS ± SD	Percentage of failure pattern		
		Adhesive	Mixed	Cohesive
1. SB + HC	16.12 ± 1.54[a]	100	0	0
2. SB + HC + AD	22.78 ± 2.44[b]	80	20	0
3. SB + HC + Si + AD	29.29 ± 2.58[c]	60	40	0
4. SB + HC + UA	28.34 ± 1.26[c]	70	30	0
5. HF + HC	10.02 ± 3.31[d]	100	0	0
6. HF + HC + AD	14.98 ± 1.56[a]	100	0	0
7. HF + HC + Si + AD	15.48 ± 2.15[a]	100	0	0
8. HF + HC + UA	15.02 ± 1.64[a]	100	0	0

Abbreviations: AD, adhesive; HF, hydrofluoric acid; MSBS, microshear bond strength; SB, sandblast; SD, standard deviation; Si, silane; UA, universal adhesive.
Note: There is no statistically significant difference when the value has the same superscript letters.

5, 6, 7, and 8 had an adhesive failure pattern after they were all broken. Furthermore, mixed failure scenarios were brought up in groups 2 to 4. Group 3 presented the greatest proportion of mixed failure patterns, accounting for 40% of the total.

Discussion

The current investigation assessed the effect of the different micromechanical surface preparations and chemically adhesive surface modification strategies applied to resin-matrix ceramics (Shofu Block HC) that were repaired using resin composites, especially using conventional ADs and UAs containing Si coupling agents. The results indicate that each group's MSBS levels differ significantly from one another. As a result, the null hypothesis was invalidated.

The current concept of adhesion frequently depends on the interaction of micromechanical retention and chemical bonding connections. It is necessary to understand how different surface modifications affect the interaction between resin-matrix ceramic materials and resin composites to create an effective connection between them. For superior micromechanical retention, the roughness of the resin-matrix ceramic surface must be created by sandblasting and HF etching.[4,11,17,18] The sandblasting protocol improved the MSBS values as compared with the non-SB group.[11] This is because applying SB greatly increases the resin-matrix ceramic's surface energy and roughness.[11,19] Alternatively, sandblasting raises the MSBS via the inorganic filler particles exposed inside the resin matrix, which in turn encourages the siloxane bond to form across the inorganic particles and the silanol within the Si coupling agent primer.[20] Providing a newly cleansed bonding region after saliva contamination is one of the additional advantages of sandblasted CAD/CAM resin.[21] In addition, the application of HF to resin-matrix ceramics did not result in a statistically significant enhancement in MSBS, inadequate surface roughness, and outcomes mostly in the adhesive mode pattern, creating an unfavorable bond, which agrees with the results of previous studies.[17,22] Furthermore, it has been noted that using HF for restoring resin-matrix ceramics might be unexpected.[17] In this investigation, it was found that the MSBS of the SB group is higher than that of the HF group. The resin-matrix ceramic surface cannot be sufficiently roughened by the HF etching. On the contrary, the SB abraded on the resin-matrix ceramic surface through pressure creates micromechanical roughness and may expose inorganic filler, and this situation for the Si coupling agent can provide chemical adhesion between resin-matrix ceramic and resin composite. As a result, the SB is the most effective protocol for producing micromechanical retention for the resin-matrix ceramic.

According to the chemically adhesive surface modifications, the HC primer is a protocol for chemically surface-treated resin-matrix ceramic (Shofu Block HC) followed by manufacturer instruction. To achieve durable adhesive bonding of resin-matrix ceramic, the HC primer is developed for use with resin-matrix ceramic CAD/CAM restorations.[23–25] The major components of HC primer are urethane dimethacrylate (UDMA) and methylmethacrylate (MMA), which together may create a thick coating of resin material at the interfaces of resin-matrix ceramic and resin composite.[23] Given that the HC primer contained UDMA and MMA, it is expected that the primer penetrated the sandblasted surface treatment of the resin-matrix ceramic and subsequently conducted curing there. The HC primer mechanism has two possible explanations: (1) the thick layer of the primer might absorb the stress from polymerization and decrease the degree of stress at the interfaces within the resin-matrix ceramic and resin composite[23]; (2) the polymer resin matrix of resin-matrix ceramic may expand as a result of the MMA; this would allow the UDMA monomer to permeate into the polymer resin matrix.[24] Moreover, when using the HC primer prior to the use of conventional AD agents, this study found that the MSBS of the HC + AD group (22.78 ± 2.44 MPa) was significantly statistically higher than the only HC-treated groups (16.12 ± 1.54 MPa). This is so that interpenetrating polymer linkages may be formed, improving the MSBS, by copolymerizing the UDMA and MMA monomers in the HC primer with a conventional AD agent monomer.[26]

For the sandblasted resin-matrix ceramic surfaces treated with HC, Si, and AD, this study demonstrated that the MSBS of the HC + Si + AD group (29.29 ± 2.58 MPa) was significantly statistically higher than the HC + AD group (22.78 ± 2.44 MPa). For the chemical adhesion process of silica-based materials, Si primer is advised because it forms a siloxane linkage on the outermost layer of ceramic and facilitates the connection between silica in the ceramic and the resin matrix.[12] The Si attaching to the unprotected SiO_2 fillers in resin-matrix ceramics may lead to an increase in the MSBS between the resin composite interface and the resin-matrix ceramic. The following are the three possible approaches that might improve the MSBS: (1) the HC primer may cause the polymer resin matrix of resin-matrix ceramic to expand by absorbing and reducing polymerization-related stress[23,24]; (2) it is possible for Si and silica particles in resin-matrix ceramics to successfully create chemical linkages[12]; and (3) it can be achievable to copolymerize the UDMA and MMA monomers in the HC primer with the conventional AD agent monomers.[26]

For the sandblasted resin-matrix ceramic surfaces treated with HC and UA, this study indicated that the MSBS of the HC + UA group (28.34 ± 1.26 MPa) was not statistically significant compared with the HC + Si + AD group (29.29 ± 2.58 MPa). The UA (Beautibond Xtreme) is composed of phosphate and carboxylate monomers and an acid-resistant Si (ARS) coupling agent. This means that UA's phosphate functional monomer may chemically attach to the zirconium particle in resin-matrix ceramic by generating a direct chemical link with the zirconium oxide.[27] Furthermore, Silva et al concluded that the phosphate functional monomer chemically bonds with the polymer matrix of resin-matrix ceramic to penetrate deeply into microretentive zones and strengthen the connection between them.[28] Prior research has documented the advantageous outcomes of resin-matrix ceramic surface treatment using UA containing Si after sandblasting.[4] Conversely, Yao et al revealed that low pH UAs containing Si may have a poor bonding ability due to the Si agents' self-condensation reaction.[29] Depending on the kind of Si used in the UA, Leelaponglit et al noticed either an increase or decrease in the bonding strength.[13] The ARS form of the Si coupling agent found in Beautibond Xtreme UA allows it to function effectively at low adhesive pH levels, protecting the Si agent from cyclic self-condensation and facilitating the Si agent's capacity to adhere to the silica particle in resin-matrix ceramic, thus improving the MSBS of resin-matrix ceramic and resin composite. The following are the four potential strategies: (1) the polymer resin matrix of resin-matrix ceramic may expand as a result of the HC primer's ability to absorb and lessen polymerization-related stress[23,24]; (2) the UDMA and MMA monomers in the HC primer may copolymerize with the UA agent monomers[26]; (3) the acidic phosphate function monomer in the UA enables chemically promoted adhesion to the zirconium particle and polymer matrix in the resin-matrix ceramic[27,28]; and (4) it is possible for the ARS and the silica particle in resin-matrix ceramics to effectively produce chemical interactions.

The findings from the evaluation of the debonded specimens' breakdown processes corresponded with the outcomes of the MSBS test. Adhesive breakdowns were more common in this study's HF groups and SB + HC (group 1), which had lower MSBS values. Mixed breakdowns appeared in the SB resin-matrix ceramic-treated groups 2 to 4, which had higher MSBS values. Low bond strength tends to be caused by adhesive failure, whereas greater adhesion is indicated by mixed failure.[26,27] It showed a clear correlation between the entire number of mixed breakdowns and bond competence; the number of mixed breakdowns increased with bond competence.

This investigation design was constrained since it focused on using a specific resin-matrix ceramic, the Shofu Block HC CAD/CAM material, making it inapplicable to other resin-matrix ceramics. It examined the incubated sample 1 day after bonding to assess the MSBS of the resin-matrix ceramic repaired with resin composites. The longevity and durability of repairs using resin composites to resin-matrix ceramic materials may be evaluated in future periods using the aging process via thermocycling. The MSBS is just one of the parameters that have been related to the performance of an adhesion technique in a dental clinical situation. In this regard, a careful review of our investigation's results is important.

Conclusion

The present in vitro research's outcomes, considering the restrictions of the research, suggested that the SB technique is the most effective protocol for producing micromechanical retention for the resin-matrix ceramic (Shofu Block HC CAD/CAM material). The application of HC primer and Si coupling agent prior to the adhesive agent is the best chemically adhesive surface modification strategy for sandblasted resin-matrix ceramic surfaces. Additionally, the application of HC primer before the use of UA containing ARS is the best alternative chemically adhesive surface modification strategy for improving the MSBS of sandblasted resin-matrix ceramic surfaces repaired with the resin composite.

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Conflict of Interest
None declared.

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