Effect of Surface Treatments on Shear Bond Strength of Resin Cement to Hybrid Ceramic Materials

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Abstract

This study evaluated the effect of chemical surface treatments on shear bond strength (SBS) of resin cement to the hybrid ceramic materials. Hybrid ceramic blocks (Shofu Block HC, Cerasmart, and Vita Enamic) were cut into slabs. The specimens of each hybrid ceramic material were randomly divided into 10 groups and each group was subdivided into 3 subgroups (n = 10) according to surface modification methods : no treatment, bonding agent (Adper Single Bond 2), Unifast TRAD Liquid + bonding agent, Meliodent Rapid Repair Liquid + bonding agent, Acetone 20 s + bonding agent, Acetone 40 s + bonding agent, Chloroform 20 s + bonding agent, Chloroform 40 s + bonding agent, Hybrid ceramic primer (HC Primer), and Hybrid ceramic primer + bonding agent. All specimens were luted using self-adhesive resin cement. SBS measurements were performed after 24 h of water storage.

Results were analyzed using two-way analysis of variance (ANOVA) and Bonferroni post hoc test ($P < 0.05$). All surface treatment methods increased the SBS of all tested materials. For Shofu Block HC and Cerasmart, the highest SBS results were obtained with Unifast TRAD Liquid + bonding agent (18.38 ± 0.73 and 18.11 ± 2.41 MPa, respectively). For Vita Enamic, HC primer + bonding agent showed the highest SBS result (20.88 ± 2.48 MPa). For scanning electron microscopy analysis, the treated surfaces showed the different morphology.

The appropriate chemical reagents could improve the SBS between resin cement and hybrid ceramic materials.

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Introduction

Computer aided design and computer aided manufacturing (CAD/CAM) technology in dentistry have been progressively developed. The design and manufacturing of dental restorations can be carried out either in a commercial dental laboratory or directly in a dental office. Compared with conventionally fabricated methods that solely rely on manual work, CAD/CAM technology reduces working time, improves the homogeneity of the restorations and raises the safety level of the manufacturing process. A wide range of CAD/CAM blocks are available for esthetic dental restorations including leucite-reinforced glass ceramics, lithium silicate/disilicate glass ceramics, zirconia-based polycrystalline ceramics, and hybrid ceramics[1,2].

New materials, resin-ceramic hybrid materials or hybrid ceramics, have been developed for CAD/CAM technique. These materials combine the advantageous properties of ceramics, such as durability and color stability, with those of composite resins, such as low rigidity, brittleness, hardness and abrasiveness[2-4]. These hybrid ceramics can be divided into 2 distinct classes based on the method of incorporation of ceramic into the polymer matrix: polymer infiltrated ceramic network and resin nanoceramic[1,5].

The adhesion and bonding of an indirect restoration to the tooth structure may prevent

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microleakage, enhance marginal adaptation, obtain high retention, and affect the longevity and success of treatment. However, these bonds depend on understanding the internal structure of the restorative material and properly selecting the suitable surface treatment and resin adhesive[6]. Therefore, to enhance the bonding, non-destructive, simple, and applicable methods that could increase surface properties of indirect restorations would be clinical beneficial[7].

One of the most common methods used to improve the mechanical retention of materials is to subject them to air particle abrasion. Several studies have shown that surface pretreatment with aluminum oxide air abrasion or tribochemical coating resulted in significant improvement in bond strength especially resin nanoceramic which is the subcategorized of hybrid ceramics [8-17]. However, air abrasion procedure requires some sensitive techniques due to a high amount of sandblasting pressure, particle sizes, or sandblasting times that could cause surface damage and resulted in a negative impact to the bond strength between luting cement and hybrid ceramic materials[11,13-15,18,19].

Numerous studies used hydrofluoric acid etching pre-treatment method to modify the microstructure of the hybrid ceramic surfaces by the dissolution of the glassy or crystalline phases, whereas the bond strength results of hydrofluoric acid etching method seemed to benefit only polymer infiltrated ceramic network (Vita Enamic) material [8-10,16,20,21].

The industrial polymerization of hybrid ceramic blocks, which has been shown a high degree of conversion and cross-linking[5]. Therefore, in an attempt to improve the bonding between resin cement and hybrid ceramic materials, chemical surface treatment that facilitates micromechanical retention has been suggested. Wetting the polymeric resin matrix surface with monomer liquid or an organic solvent such as acetone or chloroform may lead to swelling of the polymer and enhancing the micromechanical retention of the hybrid ceramic surfaces.

Hybrid ceramic primer (HC Primer, Shofu Inc., Kyoto, Japan) has been introduced to the market as a single component primer developed to treat the bonding surface of hybrid ceramics after mechanically roughened of surfaces. The manufacturer attributed that methyl methacrylate (MMA) containing in HC Primer can infiltrate the resin matrix of hybrid ceramic surfaces and enhance bonding performance of resin cement and hybrid ceramics.

Regarding the surface treatment methods of hybrid ceramics, there are no publications of research about HC Primer, monomer liquids, or organic solvents conditioning effect on the use of the bonding efficiency to different manufacturers of hybrid ceramic blocks. Therefore, this study aimed to evaluate the effect of different chemical surface treatments on shear bond strength (SBS) of self-adhesive resin cement to the hybrid ceramic materials.

The null hypothesis of this study was that different chemical surface treatments would not affect the SBS of resin cement to hybrid ceramics.

Materials and methods

Specimens preparation

The materials tested and their respective compositions are displayed in *Table 1*. Three commercial hybrid ceramics used in this study were Shofu Block HC (Shofu Inc., Kyoto, Japan), Cerasmart (GC Corp., Tokyo, Japan), and Vita Enamic (Vita Zahnfabrik H. Rauter, Bad Sackingen, Germany). From each hybrid ceramic, 100 square-shaped specimens (8 x 8 x 2 mm) were prepared from pre-fabricated blocks using a water-cooled diamond blade (Diamond Wafering Blade, Buehler Ltd, Lake Bluff, IL, USA) with a low-speed cutting saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA). The specimens were positioned in a polyvinyl chloride plastic tubes and embedded in dental gypsum type IV. The specimens were then polished under water cooling by using 600-grit silicon carbide abrasive paper for 1 minute by a polishing machine with an automatic head (Nano 2000T grinder-polisher with a FEMTO 1000 polishing head, Pace Technologies, Tucson, AZ, USA). After being polished, the samples were ultrasonically cleaned (CP360 Powersonic, Crest Ultrasonics, Ewing Township, NJ, USA) in distilled water for 5 minutes. The specimens were then etched with 35% phosphoric acid gel (Scotchbond Universal Etchant, 3M ESPE, St. Paul, MN, USA) for 15 seconds and rinsed with distilled water for 15 seconds to remove grease and they were air-dried for 10 seconds.

Three hundred-composite rods were prepared using silicone mold with 4 mm inner diameter and 5 mm height. A single increment of composite resin (Filtek Bulk Fill Posterior (A3 Shade), 3M ESPE, St. Paul, MN, USA) was filled in the silicone mold. Light curing was performed using an LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) for 40 seconds. After removal of the mold, irradiation of both sides of composite rod was repeated for 40 seconds each. The bonding surface was then air abraded by using 50 μm aluminum oxide at 0.1 MPa for 10 seconds, and subsequently, composite rods were ultrasonically cleaned in distilled water for 15 minutes.

The specimens of each hybrid ceramic material were randomly divided into 10 groups and each group was subdivided into 3 subgroups (n = 10) according to surface modification methods as shown in Figure.1. The surface modification details are shown in *Table 2*.

To standardize the bonding area of each specimen, a single-sided adhesive tape was prepared. An 80-micron thick single-sided adhesive tape (Scotch blue Painter's tape, 3M, St. Paul, MN, USA) was cut into a square shape with a size of 6x6 mm. A 3-mm diameter hole was made in the middle of the adhesive tape using a hole-puncher. The adhesive tape was placed on the surface of each specimen and the hole represents the bonding site for composite resin.
After all surface treatments, the resin cement (RelyX Unicem Aplicap, 3M ESPE, St. Paul, MN, USA) was mixed and applied to the bonding area and placed the composite rod. The cement was allowed to flow under a constant pressure of 1,000 gram on top. Then resin cement was light-cured from four sides for 40 seconds each with LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA). Specimens were stored in distilled water at 37 °C for 24 hours before testing.

Shear bond strength measurements

SBS was measured with a universal testing machine (EZ-S 500N, Shimadzu Corporation, Kyoto, Japan). The specimen was fixed in metal mounting jig and the ceramic-composite resin interface was loaded at a crosshead speed of 0.5 mm/min until fracture occurred. The force required for failure was recorded in Newton and divided by the bonding area (mm^2) to calculate the SBS in MPa. The debonded specimens were examined under a stereomicroscope (SZ 61, OLYMPUS, Tokyo, Japan) to determine the failure mode that was classified as follows: adhesive failure, mixed failure, cohesive failure within the body of resin cement, and cohesive failure within the body of hybrid ceramic.

Scanning electron microscopy (SEM) analysis

For SEM analysis of specimen surface treated, additional hybrid ceramic specimens were prepared from each group without the application of bonding agent using a gold spotter-coated (Gold sputtering unit, JEOL Ltd., Akishima, Japan) and then observed through a scanning electron microscope (JSM-IT500HR, JEOL Ltd., Tokyo, USA).

Statistical analysis

The SPSS statistical software (IBM SPSS Statics for Windows, Version 22.0. Armonk, NY, USA) was used for statistical analysis. The Shapiro-Wilk tests was performed to verify the normality of SBS data distribution. Then, the SBS results were analyzed by two-way analysis of variance (ANOVA) for evaluating the effects of hybrid ceramic material, surface treatment, and their interactions. The mean SBS values were compared by using post hoc Bonferrroni test. All statistical analysis was performed with a significant level of 0.05.

Results

The two-way ANOVA analysis revealed that the SBS was significantly influenced by the hybrid ceramic materials, surface treatment methods, and the interaction between hybrid ceramic materials and surface treatment methods (p = .001, .001, .010, F = 21.34, 40.35, 2.00, η^2 = .14, .57, .12, respectively), which the model is expected to account for 58% of the variance in the SBS (*Table 3*). Moreover, surface treatments is a moderate effect size to SBS, while, hybrid ceramic materials and their interactions are small effect size [22].

The mean SBS results and failure analysis for three hybrid ceramics were summarized in *Table 4*. In general, Vita Enamic showed higher SBS compared with Shofu Block HC and Cerasmart. Furthermore, control group of each material showed the lowest SBS value. UB and PB groups mostly showed the highest SBS values for all hybrid ceramics.

For Shofu Block HC, UB showed statistically significant higher SBS than MB, C40B, A40B, A20B, B, and control groups. Intermediate results were found for C20B, PB and P groups. For Cerasmart, UB, PB, C20B, P, and MB resulted in statistically significant higher SBS than A20B and control groups. Surface treatment in B, C40B, and A40B groups yielded intermediate SBS values. For Vita Enamic, the highest SBS values were observed in PB, UB, and P groups. These means were statistically significantly higher than those of C20B, A20B, A40B, B, and control groups. Intermediate SBS results were found for C40B and MB. The failure-mode analysis revealed a high rate of adhesive failure in the control groups for all hybrid ceramics. The mixed failure was increased as the surface treatment applied, while no cohesive failure was seen in all groups.

Representative SEM images of the treated Shofu Block HC, Cerasmart, and Vita Enamic are presented in Figure.2. From the control groups, the different hybrid ceramic materials showed the different surface morphologies. The surfaces of specimens treated by Unifast TRAD Liquid application showed the change in surface texture with several randomly distributed rod-like particles attached to the treated surfaces. For the groups treated with Unifast TRAD Liquid, Meliodent Rapid Repair Liquid, acetone and chloroform, the treated surfaces appeared to partially dissolve the polymer matrix. Increasing the application time from 20 seconds to 40 seconds, the treated surface of acetone or chloroform showed slightly different morphology. The treated surface of specimen from group 2, 9 and10 demonstrated smoother surface. Therefore, the surface morphology of treated specimens from these groups could not evaluate by SEM. Figure.3 is a SEM image of a treated Shofu Block HC specimen applied with HC primer which the irregular surface morphology disappeared.

Discussion

This study was conducted to evaluate the effect of surface treatment of hybrid ceramics with monomer liquid, organic solvents, and hybrid ceramic primer. The bonding efficacy was evaluated using the SBS test and the treated surfaces were evaluated by SEM. Besides the determination of the bond strength values, the failure modes were also examined in this study. The results of the current study revealed that the chemical surface treatment affected the SBS of resin cement to hybrid ceramics. Therefore, the findings of this study led to the rejection of the null hypothesis that there would be no difference on the shear bond strength based on chemical surface treatment methods.

The bond strength of resin cement to hybrid ceramics can be improved by the application of HC Primer, Unifast TRAD Liquid, chloroform, Meliodent Rapid Repair Liquid, and acetone followed by Adper Single Bond 2 compared with control groups, as confirmed by the failure-mode results in this study. Adhesive failures decreased and mixed failures increased in the surface treatment groups. However, cohesive failure was not presented in this study. These hybrid ceramics were expected to show less cohesive failure within the materials due to the polymer-ceramic association, which significantly decrease the material's brittleness[3,4].

The SEM analysis revealed that the surface of hybrid ceramics treated by application of Unifast TRAD Liquid, Meliodent Rapid Repair Liquid, acetone, and chloroform showed irregular and distributed gaps and pores which possibly resulted from the swelling phenomenon of polymer matrix[23]. The polymer matrix of hybrid ceramic was suffered from a selective dissolution when exposed to the solvents, promoting a better micromechanical interlocking with resin bonding. Vita Enamic and Shofu Block HC contain urethane dimethacrylate (UDMA) and triethyleneglycol dimethacrylate (TEGDMA) monomers, while Cerasmart has slight differences in monomer mixture UDMA, dimethacrylate (DMA), and 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane (Bis-MEPP). Therefore, the conditioning liquids used in the current experiment were selected for the following reasons: polymer solubility and swelling occurs when the polymer and solvent solubility parameters and polarities are close to each other. The solubility parameter of UDMA and TEGDMA (resin monomers of hybrid ceramics) are 21.3 and 19.2 MPa$^{1/2}$, respectively[24,25]. While those of MMA, acetone, and chloroform are 18.0, 19.7, and 18.5 MPa$^{1/2}$, respectively[25].

According to Shen et al.[26], the chemical surface dissolution of acrylic resins could be affected by the degree of crosslinking of the polymer chains. Additionally, Ellis and Faraj[27] stated that cross-linked acrylic resins only swell in solvents such as chloroform or acetone. However, it is not clear whether or not these principles are applicable to cross-linked hybrid ceramic materials.

Vallittu et al. [28] reported that wetting the repair surfaces with MMA dissolved the surface structure of polymethyl methacrylate (PMMA), and a duration of 180 seconds of wetting with MMA enhanced adhesion, compared with shorter durations of wetting. Therefore, in the present study the MMA treatment was selected for 180 seconds. The results of this study indicated that MMA treatment improved the bond strength of resin cement and hybrid ceramics. Unifast TRAD Liquid and Meliodent Rapid Repair Liquid also contain MMA as a monomer. However, the manufacture's confidential information was not exactly specified about the composition. UB was superior to that of MB for treating the three hybrid ceramics, due to UB exhibited significantly higher SBS values compared with MB except Cerasmart. The explanation may be the specific composition of Unifast TRAD Liquid, which was confirmed by SEM observation. SEM images of Unifast TRAD Liquid group showed superficial dissolution of resin matrix, as well as the formation of numerous rod-like particles (Figure.2B), which possibly served for micromechanical retention of resin bonding, while Meliodent Rapid Repair Liquid group revealed only the dissolution of the resin matrix (Figure.2C).

However, the application of acetone combination with Adper Single Bond 2 did not improve the bond strength between resin cement and hybrid ceramics compared with the B group. For A20B and A40B groups, the SBS results of all hybrid ceramics to resin cement were not significantly different. However, for Cerasmart, A40B group was slightly higher SBS than A20B group. It is speculated that the evaporation of acetone after the application was too quick to effectively dissolve and swell the polymer matrix, which in turn might have insufficient micromechanical retention for resin bonding.

Chloroform is an organic solvent which appears to facilitate the swelling of the polymer component. The bonding mechanisms of chloroform is likely to be the same as that of acetone groups, i.e. swelling and superficial disintegration of the hybrid ceramic surfaces by the solvent. Moreover, chloroform created irregular surfaces that were quite similar to those caused by acetone treatment. The increase of the SBS in Shofu Block HC and Cerasmart C20B groups, and Vita Enamic C40B group may be explained by SEM evaluations. SEM images of the Vita Enamic treated by chloroform for 40 seconds revealed swelling of polymer component covered most of the ceramic component (Figure.2G), while treating with chloroform for 20 seconds, the ceramic component could be seen (Figure.2F). But the slight increase of the SBS of Vita Enamic C40B group showed no significant difference from the Vita Enamic C20B group. The surfaces of the Shofu Block HC and Cerasmart pretreated with chloroform for 20 and 40 seconds presented the similar swelling of the resin matrix (Figures.2F and 2G). But the SBS in Shofu Block HC and Cerasmart C20B groups were higher than Shofu Block HC and Cerasmart C40B groups. This indicated that the application time of chloroform solvent affected the SBS of hybrid ceramic materials.

The application of bonding agent resulted in increased SBS between resin cement and hybrid ceramics, especially after the surface treatments. The bonding agent acts as a preparing agent who improved the resin bonding. The explanation may lie in the different capacities to wet the hybrid ceramic surface between the bonding agent and luting cement. The viscosity of the unfilled bonding agent Adper Single Bond 2 is considerably lower than that of RelyX Unicem resin cement. Thus, the capacity of the Adper Single Bond 2 to wet the surface and penetrate the treated surface of hybrid ceramic is greater. However, previous studies reported that the specific chemical composition of universal adhesives (such as silane, MDP (10methacryloyloxydecyl-dihydrogen phosphate), methacrylate monomer, and chlorhexidine) was not the decisive factor in the bond strength between resin cement and CAD/CAM restorative materials [29].

Barutcigil et al. [8] founded that the using bonding agent without the surface treatment improved SBS of Vita Enamic hybrid ceramic. Similarly, in our study, the SBS of Adper Single Bond 2 group was higher than that of the control group. Moreover, other studies reported that universal adhesive proved to be effective intermediate agents after surface roughening of hybrid ceramic materials[12,19,21,30]. Komurcuoglu et al.[21] revealed that hydrofluoric acid etching or sandblasting in combination with a bonding agent pretreatment was more effective than mechanical surface treatment alone for Vita Enamic and Lava Ultimate.

HC Primer is commercially available conditioning liquid, which was specifically developed to improve the adhesion to hybrid ceramic materials, using a combination of MMA, acetone, and UDMA. HC Primer was selected for comparison between the commercial hybrid ceramic primer and common monomer liquid and organic solvents that used in a clinical situation. In the present study, P group showed high bond strength values, which comparable to UB, MB, C40B, and C20B groups. Moreover, PB groups presented the superior enhancement of the bond strength compared with P groups. Although the mechanism of action of HC Primer is unclear, the rationale behind the composition of HC Primer was that the MMA (10-20 wt.%) and acetone (10-20 wt.%) were regarded appropriate to swell the polymer surface and in turn to increase its permeability for the penetration of the UDMA.

The choice of resin cements in luting indirect restorations is a critical factor. Self-adhesive resin cements have widely been used for the last decades. Self-adhesive resin cements do not have any pretreatment procedure on the tooth surface. Moreover, ease of handling property overcame the technique sensitivity of luting cement. Luting cement application is known to be complicated because this application is technical demanding and sensitive[31]. Bellan et al.[32] reported that the self-adhesive resin cement was comparable to conventional resin cement with total-etch or self-etch adhesive systems in the bond of CAD/CAM hybrid ceramic materials to dentin. In this study, self-adhesive resin cement was considered as the luting agent because of its widespread use.

This study had limitations that should be mentioned. Further studies should be conducted to investigate other factors involved in the combination of solvents with varies application periods, various adhesive resins, the aging process and thermo-cycling test and the evaluation of different cementation strategies.

Conclusions

The summary of the results points to the fact that chemical surface treatments are alternative surface treatment methods to achieve a proper adhesion between resin cement and hybrid ceramic materials. The application of MMA or chloroform in combination with bonding agent resulted in comparable SBS results to commercial hybrid ceramic primer.

Declaration of Interest

The authors report no conflict of interest.

Material	Manufacturer	Composition	Batch No.
Shofu Block HC	Shofu Inc., Kyoto, Japan	Silica powder, micro fumed silica, zirconium silicate, UDMA, TEGDMA	071601
Cerasmart	GC Corp., Tokyo, Japan	Silica (20 nm), barium glass (300 nm), Bis-MEPP, UDMA, DMA	1706151
Vita Enamic	Vita Zahnfabrik H. Rauter, Bad Sackingen, Germany	Feldspar ceramic enriched with aluminum oxide, UDMA, TEGDMA	62430
Filtek Bulk Fill Posterior	3M ESPE, St. Paul, MN, USA	Aromatic UDMA, UDMA, 1,12-dodecanedimethacrylate, non-agglomerated/non-aggregated silica filler, non-agglomerated/nonaggregated filler, aggregated zirconia/silica cluster filler, ytterbium trifluoride filler	N956965
Adper Single Bond 2	3M ESPE, St. Paul, MN, USA	Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly(itaconic) acid, 10% by weight of 5 nm-diameter spherical silica particles	N825173
HC primer	Shofu Inc., Kyoto, Japan	10-20% MMA, 10-20% acetone, UDMA, polymerization initiator and others	011707
Unifast TRAD Liquid	GC America Inc., Alsip, IL, USA	90% MMA, 1-5% N, N-dimethyl-p-toluidine, 0.5-1% 2-(2H-benzotriazol-2-yl)-p-cresol, 0.1-0.5% EGDMA	1709111
Meliodent Rapid Repair Liquid	Heraeus Kulzer GmbH, Hanau, Germany	90% MMA, <1% N, N-dimethyl-p-toluidine, <1% 2-(2H-benzotriazol-2-yl)-4-methylphenol, 0-5% tetramethylene dimethacrylate	020104
Acetone	EMSURE®, Merck KGaA, Darmstadt, Germany	100% Acetone	K49984814 809
Chloroform	RCI Labscan Limited, Bangkok, Thailand	99% Chloroform, 1% ethanol	18020122
RelyX Unicem Aplicap	3M ESPE, St. Paul, MN, USA	Powder: oxide glass chemicals (non-fibrous), silane treated silica, substituted pyrimidine, calcium hydroxide, sodium persulfate, titanium dioxide Liquid: mixture of mono-, di-, and tri-glycerine dimethacrylate ester of phosphoric acid, TEGDMA, substituted dimethacrylate, copper acetate	4178616

Table 1. Materials used in this study.
Bis-MEPP, 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane; Bis-GMA, bisphenol A diglycidylether dimethacrylate; DMA, dimethacrylate; EGDMA, ethylene glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MMA, methyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

Group	Code	Surface modification details
1	C (Control)	No surface treatment
2	B	Two microliters of bonding agent (Adper Single Bond 2, 3M ESPE, St. Paul, MN, USA) were applied to the specimen surface using micropipette (Eppendorf AG, Hamburg, Germany) and the surface was rubbed with a disposable microbrush (Citisen Micro Applicator, Huanghua Promisee Dental, Hebei, China) for 10 sec. The excess bonding agent was removed with a new disposable microbrush, then air-dried for 20 sec and light activated for 40 sec.
3	UB	Two microliters of Unifast TRAD Liquid (GC America Inc., Alsip, IL, USA) were applied to the specimen surface each time for 3 rounds. Each round the surface was rubbed with a new disposable microbrush for 10 sec and left undisturbed for 50 sec. After the third application, the treated surface was air-dried for 20 sec. The bonding agent was applied as described in group 2.
4	MB	Two microliters of Meliodent Rapid Repair Liquid (Heraeus Kulzer GmbH, Hanau, Germany) were applied to the specimen surface each time for 3 rounds. Each round the surface was rubbed with a new disposable microbrush for 10 sec and left undisturbed for 50 sec. After the third application, the treated surface was air-dried for 20 sec. The bonding agent was applied as described in group 2.
5	A20B	Two microliters of acetone (EMSURE®, Merck KGaA, Darmstadt, Germany) were applied to the specimen surface each time for 2 rounds. Each round the surface was rubbed with a new disposable microbrush for 10 sec. After the second application, the treated surface was air-dried for 20 sec. The bonding agent was applied as described in group 2.
6	A40B	Two microliters of acetone were applied to the specimen surface each time for 4 rounds. Each round the surface was rubbed with a new disposable microbrush for 10 sec. After the fourth application, the treated surface was air-dried for 20 sec. The bonding agent was applied as described in group 2.
7	C20B	Two microliters of chloroform (RCI Labscan Limited, Bangkok, Thailand) were applied to the specimen surface each time for 2 rounds. Each round the surface was rubbed with a new disposable microbrush for 10 sec. After the second application, the treated surface was air-dried for 20 sec. The bonding agent was applied as described in group 2.
8	C40B	Two microliters of chloroform were applied to the specimen surface each time for 4 rounds. Each round the surface was rubbed with a new disposable microbrush for 10 sec. After the fourth application, the treated surface was air-dried for 20 sec. The bonding agent was applied as described in group 2.
9	P	Two microliters of hybrid ceramic primer (HC Primer, Shofu Inc., Kyoto, Japan) were applied to the specimen surface. The excess primer was removed with a new disposable microbrush, then air-dried for 20 sec and light activated for 10 sec.
10	PB	Hybrid ceramic primer was applied as described in group 9, followed by bonding agent as described in group 2.

Table 2. Summary of surface modification details for each group.
Group 1, 2, 9, and 10. The adhesive tape with a central hole was firmly attached to the specimen surface before any chemical application. Group 3-8. After chemical treatment, the adhesive tape with a central hole was firmly attached to the specimen surface before the bonding agent application step.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared (η^2)
Hybrid ceramic	162.11	2	81.06	21.34	.001	.136
Surface treatment	1379.47	9	153.27	40.35	.001	.574
Hybrid ceramic * Surface treatment	136.90	18	7.61	2.00	.010	.118
Error	1025.66	270	3.80			
Total	2704.14	299				

Table 3. Results of two-way ANOVA test.
a. R Squared = .621 (Adjusted R Squared = .580)

Group	Shofu Block HC		Cerasmart		Vita Enamic	
	Mean SBS ± SD	Fracture mode	Mean SBS ± SD	Fracture mode	Mean SBS ± SD	Fracture mode
C	11.18 ± 2.05^{aA}	[100/0/0/0]	10.76 ± 2.23^{aA}	[100/0/0/0]	12.69 ± 2.32^{aA}	[100/0/0/0]
B	13.10 ± 0.91^{abA}	[90/10/0/0]	13.66 ± 2.07^{bcAB}	[80/20/0/0]	15.36 ± 2.38^{abB}	[80/20/0/0]
UB	18.38 ± 0.73^{dA}	[60/40/0/0]	18.11 ± 2.41^{eA}	[40/60/0/0]	19.61 ± 2.78^{deA}	[70/30/0/0]
MB	15.06 ± 1.90^{bcA}	[70/30/0/0]	16.14 ± 1.84^{cdeA}	[60/40/0/0]	16.30 ± 2.76^{bcA}	[80/20/0/0]
A20B	13.87 ± 1.48^{abAB}	[90/10/0/0]	13.14 ± 2.12^{bB}	[70/30/0/0]	15.79 ± 2.60^{bA}	[50/50/0/0]
A40B	13.92 ± 1.97^{abA}	[80/20/0/0]	15.18 ± 1.90^{bcdA}	[60/40/0/0]	15.72 ± 1.58^{bA}	[60/40/0/0]
C20B	17.88 ± 1.33^{cdA}	[70/30/0/0]	17.79 ± 1.44^{deA}	[60/40/0/0]	15.97 ± 1.84^{bA}	[60/40/0/0]
C40B	14.37 ± 1.92^{bA}	[60/40/0/0]	14.80 ± 1.72^{bcAB}	[60/40/0/0]	16.75 ± 1.36^{bcdB}	[70/30/0/0]
P	15.96 ± 1.37^{bcdA}	[70/30/0/0]	16.31 ± 1.28^{cdeA}	[90/10/0/0]	19.05 ± 2.14^{cdeB}	[60/40/0/0]
PB	17.79 ± 1.79^{cdA}	[80/20/0/0]	17.89 ± 1.84^{deA}	[70/30/0/0]	20.88 ± 2.48^{eB}	[80/20/0/0]

Table 4. Mean shear bond strength values (MPa ± SD) and Number (%) of specimens according to fracture mode.
Mean values represented with same superscript uppercase letters (row) or lowercase letters (column) are not significantly to Bonferroni post hoc test (P > 0.05). Percentage of fracture mode [adhesive failure/mixed failure/cohesive failure within the body of resin cement/cohesive failure within the body of hybrid ceramic].

Figure 1. Schematic diagram of experimental procedure.

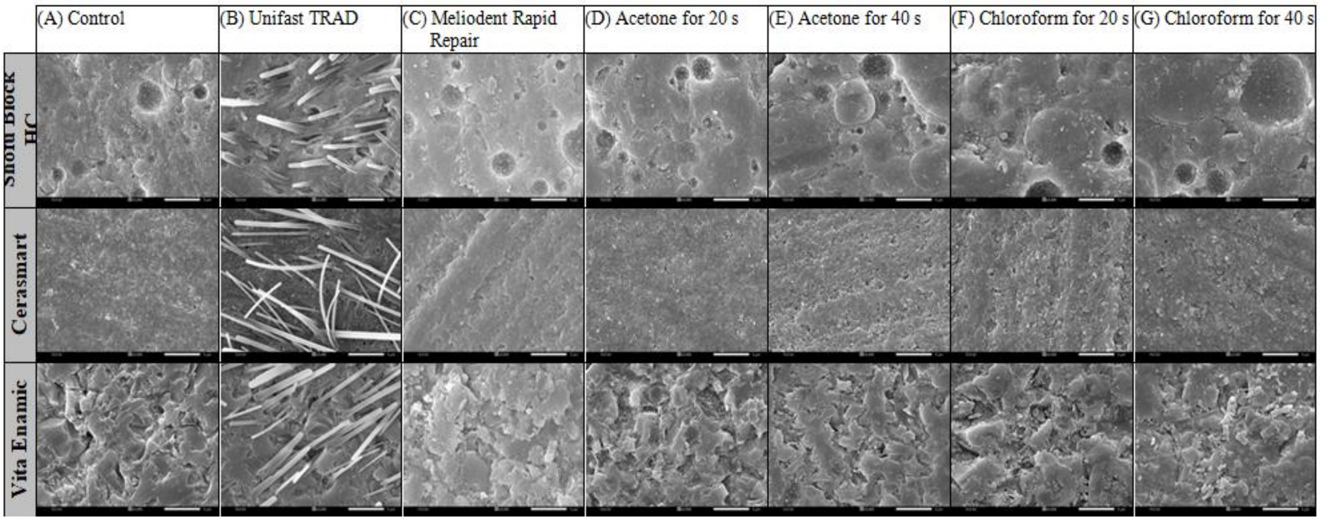

Figure 2. SEM images of the surfaces of the Shofu Block HC, Cerasmart, and Vita Enamic specimens after different surface treatments at 5,000x magnification.

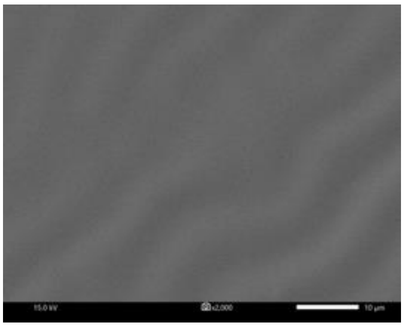

Figure 3. SEM image of the surface of the Shofu Block HC applied with HC primer at 2,000x magnification.

References

1. Duarte S, Sartori N, Phark J-H. Ceramic-Reinforced Polymers: CAD/CAM Hybrid Restorative Materials. Curr Oral Health Rep 2016;3(3):198-202.
2. Lauvahutanon S, Takahashi H, Shiozawa M, Iwasaki N, Asakawa Y, Oki M, et al. Mechanical properties of composite resin blocks for CAD/CAM. Dent Mater J 2014;33(5):705-710.
3. Della Bona A, Corazza PH, Zhang Y. Characterization of a polymer-infiltrated ceramic-network material. Dent Mater 2014;30(5):564-569.
4. Awada A, Nathanson D. Mechanical properties of resin-ceramic CAD/CAM restorative materials. J Prosthet Dent 2015;114(4):587-593.
5. Mainjot AK, Dupont NM, Oudkerk JC, Dewael TY, Sadoun MJ. From Artisanal to CAD-CAM Blocks: State of the Art of Indirect Composites. J Dent Res 2016;95(5):487-495.
6. El-Damanhoury HM, Gaintantzopoulou MD. Self-etching ceramic primer versus hydrofluoric acid etching: Etching efficacy and bonding performance. J Prosthodont Res 2018;62(1):75-83.
7. Piascik JR, Wolter SD, Stoner BR. Development of a novel surface modification for improved bonding to zirconia. Dent Mater 2011;27(5):e99-105.
8. Barutcigil K, Barutcigil Ç, Kul E, Özarslan MM, Buyukkaplan US. Effect of Different Surface Treatments on Bond Strength of Resin Cement to a CAD/CAM Restorative Material. J Prosthodont 2019;28(1):71-78.
9. Campos F, Almeida CS, Rippe MP, de Melo RM, Valandro LF, Bottino MA. Resin Bonding to a Hybrid Ceramic: Effects of Surface Treatments and Aging. Oper Dent 2016;41(2):171-178.
10. Frankenberger R, Hartmann VE, Krech M, Krämer N, Reich S, Braun A, et al. Adhesive luting of new CAD/CAM materials. Int J Comput Dent 2015;18(1):9-20.
11. Kim JE, Kim JH, Shim JS, Roh BD, Shin Y. Effect of air-particle pressures on the surface topography and bond strengths of resin cement to the hybrid ceramics. Dent Mater J 2017;36(4):454-460.
12. Park JH, Choi YS. Microtensile bond strength and micromorphologic analysis of surface-treated resin nanoceramics. J Adv Prosthodont 2016;8(4):275-284.
13. Tekçe N, Tuncer S, Demirci M. The effect of sandblasting duration on the bond durability of dual-cure adhesive cement to CAD/CAM resin restoratives. J Adv Prosthodont 2018;10(3):211-217.
14. Tekçe N, Tuncer S, Demirci M, Kara D, Baydemir C. Microtensile Bond Strength of CAD/CAM Resin Blocks to Dual-Cure Adhesive Cement: The Effect of Different Sandblasting Procedures. J Prosthodont 2019;28(2):e485-490.
15. Yoshihara K, Nagaoka N, Maruo Y, Nishigawa G, Irie M, Yoshida Y, et al. Sandblasting may damage the surface of composite CAD–CAM blocks. Dent Mater 2017;33(3):e124-135.
16. Güngör MB, Nemli SK, Bal BT, Ünver S, Doğan A. Effect of surface treatments on shear bond strength of resin composite bonded to CAD/CAM resin-ceramic hybrid materials. J Adv Prosthodont 2016;8(4):259-266.

17. Kimura F, Komine F, Kubochi K, Yagawa S. Bond strength of CAD/CAM-manufactured composite resin and ceramic veneers to a zirconia framework. J Oral Sci 2019;61(2):327-334.
18. Strasser T, Preis V, Behr M, Rosentritt M. Roughness, surface energy, and superficial damages of CAD/CAM materials after surface treatment. Clin Oral Investig 2018;22(8):2787-2797.
19. Stawarczyk B, Krawczuk A, Ilie N. Tensile bond strength of resin composite repair in vitro using different surface preparation conditionings to an aged CAD/CAM resin nanoceramic. Clin Oral Investig 2015;19(2):299-308.
20. Elsaka SE. Bond strength of novel CAD/CAM restorative materials to self-adhesive resin cement: the effect of surface treatments. J Adhes Dent 2014;16(6):531-540.
21. Komurcuoglu MB, Sagirkaya E, Tulga A. Influence of different surface treatments on bond strength of novel CAD/CAM restorative materials to resin cement. J Adv Prosthodont 2017;9(6):439-446.
22. Cohen J. Statistical Power Analysis. Curr Dir Psychol Sci 1992;1(3):98-101.
23. Vallittu PK, Ruyter IE, Nat R. The swelling phenomenon of acrylic resin polymer teeth at the interface with denture base polymers. J Prosthet Dent 1997;78(2):194-199.
24. Breschi M, Fabiani D, Sandrolini L, Colonna M, Sisti L, Vannini M, et al. Electrical properties of resin monomers used in restorative dentistry. Dent Mater 2012;28(9):1024-1031.
25. Pashley DH, Tay FR, Carvalho RM, Rueggeberg FA, Agee KA, Carrilho M, et al. From dry bonding to water-wet bonding to ethanol-wet bonding. A review of the interactions between dentin matrix and solvated resins using a macromodel of the hybrid layer. Am J Dent 2007;20(1):7-20.
26. Shen C, Colaizzi FA, Birns B. Strength of denture repairs as influenced by surface treatment. J Prosthet Dent 1984;52(6):844-848.
27. Ellis B, Faraj SA. The structure and surface topography of acrylic denture base materials. J Dent 1980;8(2):102-108.
28. Vallittu PK, Lassila VP, Lappalainen R. Wetting the repair surface with methyl methacrylate affects the transverse strength of repaired heat-polymerized resin. J Prosthet Dent 1994;72(6):639-643.
29. Siqueira F, Cardenas AM, Gutierrez MF, Malaquias P, Hass V, Reis A, et al. Laboratory Performance of Universal Adhesive Systems for Luting CAD/CAM Restorative Materials. J Adhes Dent 2016;18(4):331-340.
30. Bunchansakul N, Thamronganankul N. The effect of surface treatment on shear bond strength ofresin matrix ceramics and dual cure resin cement. J Int Dent Med Res 2020;13(4):1248-1257.
31. Ferracane JL, Stansbury JW, Burke FJ. Self-adhesive resin cements-chemistry, properties and clinical considerations. J Oral Rehabil 2011;38(4):295-314.
32. Bellan MC, Cunha PFJSd, Tavares JG, Spohr AM, Mota EG. Microtensile bond strength of CAD/CAM materials to dentin under different adhesive strategies. Braz Oral Res 2017;31(1):e109.