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Research Article

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Influence of different universal adhesives on the repair performance of hybrid CAD-CAM materials

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ABSTRACT

Objectives: The aim of this study was to investigate the microshear bond strength (µSBS) of different universal adhesive systems applied to hybrid computer-aided design/computer-aided manufacturing (CAD-CAM) restorative materials repaired with a composite resin.
Materials and Methods: Four types of CAD-CAM hybrid block materials—Lava Ultimate (LA), Vita Enamic (VE), CeraSmart (CS), and Shofu Block HC (SH)—were used in this study, in combination with the following four adhesive protocols: 1) control: porcelain primer + total etch adhesive (CO), 2) Single Bond Universal (SB), 3) All Bond Universal (AB), and 4) Clearfil Universal Bond (CU). The µSBS of the composite resin (Clearfil Majesty Esthetic) was measured and the data were analyzed using two-way analysis of variance and the Tukey test, with the level of significance set at $p < 0.05$.
Results: The CAD-CAM block type and block-adhesive combination had significant effects on the bond strength values ($p < 0.05$). Significant differences were found between the following pairs of groups: VE/CO and VE/AB, CS/CO and CS/AB, VE/CU and CS/CU, and VE/AB and CS/AB ($p < 0.05$).
Conclusions: The µSBS values were affected by hybrid block type. All tested universal adhesive treatments can be used as an alternative to the control treatment for repair, except the AB system on VE blocks (the VE/AB group). The µSBS values showed variation across different adhesive treatments on different hybrid CAD-CAM block types.

Keywords: Dental restoration repair; Computer-aided design; Dental bonding; Adhesives

INTRODUCTION

Aesthetic restorations made chair-side or in a laboratory environment using computer-aided design/computer-aided manufacturing (CAD-CAM) technology are increasingly popular treatments in contemporary dentistry [1]. A wide variety of CAD-CAM materials, such as lithium disilicate, feldspar, and resin nanoceramics, can be found on the market for different indications. Hybrid ceramics that comprise a feldspar ceramic network are combined with a polymer network [2]. The relatively new category of hybrid ceramics is also becoming increasingly diverse. These new hybrid ceramics resist chipping and cracking during milling

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and are less brittle than glass ceramics [3-5]; the other advantages of hybrid ceramics are that they do not need post-firing, they are easily polished, and they allow easier completion of restorations [6].

In the oral environment, restorations remain susceptible to fractures. Internal defects, parafunctional habits, inadequate occlusal contact, and trauma can cause indirect restorations to fracture [7,8]. Direct composite repair techniques have been proposed to avoid the disadvantages of entirely replacing a restoration, such as additional preparation, extended restoration size, other expenses, and chair-time [9,10]. Currently, multiple repair systems with various conditioning protocols and different adhesive systems are commercially available. Universal adhesives are simplified systems that usually contain all bonding components in a single bottle [11-13]. They can be applied either in etch-and-rinse or self-etching bonding protocols, according to the manufacturer's instructions. Some universal adhesives may contain silane, which eliminates the silanization step when bonding to glass ceramics, hybrid materials, and resin composites. Studies of different repair systems and protocols have been conducted [14-16]; however, there is a lack of information about the effects of universal adhesive systems on the repair performance of hybrid CAD-CAM materials.

Therefore, the aim of this study was to investigate the repair performance of different universal adhesive systems applied to four commercially available hybrid CAD-CAM restorative materials through microshear bond strength (μSBS) testing. The null hypotheses tested were that 1) the type of applied adhesive material, 2) the type of hybrid CAD-CAM block, and 3) the adhesive-block combination would not influence the μSBS values.

MATERIALS AND METHODS

Specimen preparation

Four types of CAD-CAM hybrid block materials—Lava Ultimate (LA; 3M ESPE, St. Paul, MN, USA), Vita Enamic (VE; Vita Zahnfabrik, Bad Sackingen, Germany), CeraSmart (CS; GC America, Alsip, IL, USA), and Shofu Block HC (SH; Shofu Dental Corp., San Marcos, CA, USA)—were used in this study, in combination with four different adhesive protocols for repair with a resin composite (Clearfil Majesty Esthetic A2, Kuraray, Tokyo, Japan). The brands, manufacturers, chemical compositions, and the application procedures of the materials are listed in **Table 1**.

The hybrid blocks were cut under water-cooling (Micracut 201, Metkon, Turkey) into specimens of 15 mm (length) × 15 mm (width) × 3 mm (thickness) (for each material, $n = 44$ specimens; in total, 176 specimens) with a low-speed cutting wheel and then embedded in a self-cured acrylic resin (Vertex Self Curing, Vertex Dental, Zeist, Netherlands). All specimens were aged together for 5,000 thermal cycles between 5°C and 55°C with a dwelling time of 30 seconds in each bath (Thermocycler THE-1100, SD Mechatronik, Feldkirchen-Westerham, Germany).

According to the porcelain and composite repair protocols described by Loomans and Özcan [17], the top surfaces of the specimens were roughened and air-abraded. Coarse-flat diamond burs (909H FG 806 314 068 544 040, Meisinger, Germany) were applied with two back-and-forth strokes for a total of 3 seconds using a high-speed hand-piece under air and water-cooling. A new bur was used for each specimen. Thereafter, specimens were cleaned with water and air-dried. The top surfaces of the specimens were air-abraded and silica-coated

Table 1. Materials used in the study

Product	Abbreviation	Chemical composition	Application	Manufacturer
CAD/CAM hybrid restorative material				
Lava Ultimate	LA	UDMA, resin nanoceramic containing 79 wt% nanoceramic particles	-	3M ESPE, St. Paul, MN, USA
Vita Enamic	VE	Ceramic part (86 wt%/75 vol%): SiO_2, Al_2O_3, Na_2O, K_2O, B_2O_3, ZrO_2, CaO Polymer part (14 wt%/25 vol%): UDMA, TEGDMA	-	Vita Zahnfabrik, Bad Säckingen, Germany
CeraSmart	CS	bis-MEPP, UDMA, DMA, silica (20 nm), barium glass (300 nm)	-	GC America, Alsip, IL, USA
Shofu Block HC	SH	UDMA, TEDGMA, 61 vol% silica powder, micro-fumed silica, zirconium silicate	-	Shofu Dental Corporation, San Marcos, CA, USA
Repair system and application protocol				
Single Bond Universal	SB	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	Applied the one-coat adhesive with a disposable applicator to the entire sample surface, rubbed it in for 20 sec, and air-dried the solvent with an air syringe for 5 sec. Light-cured for 10 sec.	3M ESPE, St. Paul, MN, USA
All Bond Universal	AB	MDP, bis-GMA, HEMA, ethanol, water, initiators	Applied one-coat of the adhesive with a disposable applicator to the entire sample surface, rubbed it in for 20 sec, and air-dried the solvent with an air syringe for 10 sec. Light-cured for 10 sec.	Bisco Inc., Schaumburg, IL, USA
Clearfil Universal	CU	10-MDP, bis-GMA, HEMA, ethanol, hydrophilic aliphatic dimethacrylate, colloidal silica, camphorquinone, silane coupling agent, accelerators, initiators, water	Applied one-coat of the adhesive with a disposable applicator to the entire sample surface, rubbed it in for 10 sec, and air-dried the solvent with an air syringe for 5 sec. Light-cured for 10 sec.	Kuraray, Okayama, Japan
Porcelain Primer	CO (control)	Silane with ethanol and acetone	Applied one-coat of the porcelain primer with disposable applicator to the entire sample surface, allowed to dwell for 30 sec, and air-dried the solvent with an air syringe.	Bisco Inc., Schaumburg, IL, USA
Adper Single Bond-2 (Total-Etch adhesive)		bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly(itaconic) acids, 10% by weight of 5-nm-diameter spherical silica particles	Applied one-coat of the adhesive with a disposable applicator to the entire sample surface, rubbed it in for 15 sec, and air-dried the solvent with an air syringe for 5 sec. Light-cured for 10 sec.	3M ESPE, St. Paul, MN, USA

CAD, computer-aided design; CAM, computer-aided manufacturing; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; bis-MEPP, bisphenol-A-ethoxylate(2)dimethacrylate; DMA, N,N- dimethylacrylamide; MDP, 10-methacryloyloxydecyldihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; bis-GMA, bisphenol A-glycidyl methacrylate.

(30 µm, CoJet, 3M ESPE, Seefeld, Germany) for 10 seconds at a distance of 10 mm (90°) and 2.8 bar air pressure. Loose particles were air-blown. Then, 44 specimens of each material were randomly divided into four groups for adhesive system repair ($n = 11$): 1) control (CO), porcelain primer + total etch adhesive; 2) Single Bond Universal (SB); 3) All Bond Universal (AB); 4) CU, Clearfil Universal Bond. The application steps are described in **Table 1**.

For the repair procedure, the specimens were positioned into a custom-fabricated Teflon bonding clamp and bonding mold with an inner diameter of 0.76 mm and a height of 2 mm. The repair composite (Clearfil Majesty Esthetic) was adhered onto the specimen surfaces in a 2-mm-thick layer and light-cured for 20 seconds by applying the curing unit directly onto the Teflon cylinder.

Light polymerization was performed with a light-emitting diode-curing unit (SDI Radii Plus, SDI Limited, Victoria, Australia). The light intensity was maintained above 1,000 mW/cm^2 (Hilux Ledmax curing lightmeter, Benlioglu Dental, Ankara, Turkey). All specimens were then subjected to an additional thermal cycling procedure (5,000 cycles between 5°C and 55°C; dwelling time, 30 seconds; transfer time, 10 seconds). After thermal cycling, all specimens were air-dried for 3 seconds, and kept under dry conditions.

µSBS and failure analysis

Bond strength was tested immediately after thermal cycling with a universal testing machine (Z010, Zwick, Ulm, Germany). A shear force was applied to the adhesive interface through a chisel-shaped loading device at a crosshead speed of 1 mm/min. The load at debonding was recorded, and the µSBS (σ) was calculated using the load at failure F (N) and the adhesive area A (mm^2) as $\sigma = F/A$.

The debonded area was examined for failure mode analysis with a stereomicroscope at ×25 magnification (M3Z, Leica Microsystems, Wetzlar, Germany). The failure mode was classified as cohesive failure exclusively in the block, cohesive failure exclusively in the resin composite, adhesive failure at the interface, and mixed failure at the adhesive/block interface, which included cohesive failure of the block and/or resin composite and adhesive material.

Statistical analysis

The µSBS (MPa) data were analyzed using the Kolmogorov-Smirnov and Shapiro-Wilk tests to check the normal distribution of the data. A normal distribution was found in all subgroups. Two-way analysis of variance (ANOVA) followed by the Tukey *post hoc* test was performed, with the level of significance set at $p < 0.05$. (Prism Version 6, GraphPad, La Jolla, CA, USA).

RESULTS

The results of two-way ANOVA showed that main effect of the 'adhesive' variable was not significant, but those of the 'block' variable ($p = 0.0029$) and the interaction effect ($p = 0.0162$) on µSBS values were significant (**Tables 2** and **3**). The lowest bond strength was obtained from the VE/AB group (5.94 ± 3.86 MPa), while the highest bond strength was obtained from the CS/AB group (19.32 ± 9.46 MPa).

Considering the adhesive treatment protocols for the same block type, there were no significant differences in the CO and SB treatments. In the CU adhesive treatment protocol, the CS/CU group showed a significant difference from the VE/CU and SH/CU groups ($p < 0.05$).

Table 2. Microshear bond strength values (MPa, $n = 11$) obtained from combinations of hybrid CAD/CAM blocks and universal adhesive systems

Block	Adhesive protocol			
	CO	CU	AB	SB
VE	15.58 (6.937)^{Aa}	8.988 (6.619)^{ABa}	5.942 (3.856)^{Ba}	11.06 (6.459)^{ABa}
LA	15.17 (7.871)^{Aa}	12.23 (6.915)^{Aab}	14.26 (7.149)^{Aab}	18.17 (10.31)^{Aa}
SH	11.11 (5.969)^{Aa}	9.234 (6.390)^{Aa}	13.17 (7.224)^{Aab}	12.77 (8.161)^{Aa}
CS	10.31 (5.896)^{Aa}	18.23 (11.20)^{ABb}	19.32 (9.462)^{Bb}	15.15 (10.56)^{ABa}

The values with different uppercase superscript letters in the same row and those with different lowercase superscript letters in the same column were significantly different ($p < 0.05$).
CAD, computer-aided design; CAM, computer-aided manufacturing; CO, control; CU, Clearfil Universal; AB, All Bond Universal; SB, Single Bond Universal; VE, Vita Enamic; LA, Lava Ultimate; SH, Shofu Block HC; CS, CeraSmart.

Table 3. Effects of the 'hybrid CAD/CAM block' and 'universal adhesive system' variables and their interaction effects analyzed using two-way ANOVA

Variable	SS	DF	MS	F (DFn, DFd)	*p* value
Adhesive	99.64	3	33.21	F (3, 160) = 0.5466	0.6511
Block	885.8	3	295.3	F (3, 160) = 4.860	0.0029
Interaction	1,285	9	142.8	F (9, 160) = 2.350	0.0162
Residual	9,722	160	60.76	-	-

CAD, computer-aided design; CAM, computer-aided manufacturing; ANOVA, analysis of variance; SS, sum of squares; DF, degrees of freedom; MS, mean sum of squares.

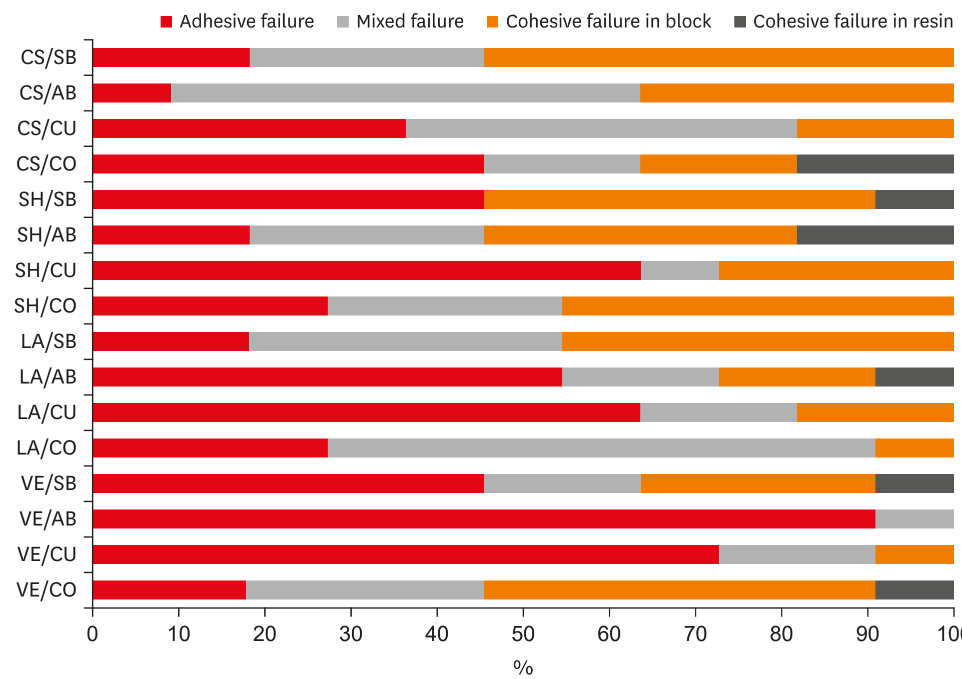

Figure 1. Distribution of fracture modes in the different combinations of hybrid CAD/CAM blocks and adhesive systems. CS, CeraSmart; SB, Single Bond Universal; AB, All Bond Universal; CU, Clearfil Universal; CO, control; SH, Shofu Block HC; LA, Lava Ultimate; VE, Vita Enamic; CAD, computer-aided design; CAM, computer-aided manufacturing.

There were no significant differences between the VE/CU, SH/CU, and LA/CU groups. In the AB adhesive treatment protocol, the CS/AB group showed a significant difference from the VE/AB group ($p < 0.05$). There were no significant differences between the VE/AB, SH/AB, and LA/AB groups.

In the analysis of different block types for the same adhesive protocol, there were no significant differences in the LA and SH blocks. In the VE and CS blocks, the control adhesive treatment, VE/CO and CS/CO groups were significantly different from the VE/AB and CS/AB adhesive treatment groups ($p < 0.05$). There were no significant differences between the VE/CU, VE/AB, and VE/SB groups. In the CS blocks, there were no significant difference between the CS/CU, CS/AB, and CS/SB groups.

Stereomicroscopic analysis

The distribution of the failure modes is shown in **Figure 1**. Adhesive failure was the predominant failure mode observed for the VE/AB, VE/CU, LA/CU, and SH/CU groups (91%, 71%, 64%, and 64%, respectively). Cohesive failure in the block was the predominant failure mode observed for the CS/SB, SH/CO, LA/SB, and VE/CO groups (54%, 45%, 45%, and 45%, respectively). Mixed failure was the predominant failure mode observed for the CS/AB, CS/CU, and LA/CO groups (54%, 45%, and 64%, respectively).

DISCUSSION

The results of this study led to the rejection of two of the null hypotheses, as the hybrid block types and the block type-adhesive treatment combinations tested in this study showed different bond strengths. However, the other null hypothesis was accepted, since the different adhesive treatments showed no differences in bond strength.

Hybrid CAD/CAM blocks consist of an organic matrix, highly filled with ceramic particles [18]. These materials are predominantly formed (> 50% by weight) of refractory inorganic compounds, regardless of the presence of a less predominant organic phase (polymer). Presently, hybrid ceramic materials can be divided into several subfamilies [19], according to their inorganic composition, such as resin nanoceramics (*e.g.*, LA and CS), glass ceramics in a resin interpenetrating matrix (*e.g.*, VE), and zirconia-silica ceramics in a resin interpenetrating matrix (*e.g.*, SH). Of the block types analyzed in this study, CS blocks showed better repair performance than VE blocks when used with the CU and AB adhesive protocols, and CS blocks showed better repair performance than SH blocks when used with the CU adhesive protocol. These differences might be related to the effects of the surface microstructure of these hybrid blocks.

Although different adhesive treatment effects showed no difference in this study, the block type-adhesive treatment combination had a significant effect. For instance, within the VE blocks, the VE/AB group (5.94 ± 3.86 MPa) showed significantly less bond strength than the VE/CO group (15.58 ± 6.93 MPa). VE, which is classified as a glass ceramic in a resin-interpenetrating matrix, is typically composed of a dual network: a feldspathic ceramic network (86% by weight, 75% by volume) and a polymer network (14% by weight, 25% by volume). The specific composition of the ceramic parts are 58% to 63% SiO_2, 20% to 23% Al_2O_3, 9% to 11% Na_2O, 4% to 6% K_2O, 0.5% to 2% B_2O_3, and less than 1% of Zr_2O and CaO. The polymer network is composed of urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA). The reason for the lower bond strength might be related to the similarity of VE to feldspathic ceramics and the silane-free composition of the AB adhesive. Higher bond strength values were observed with CO treatment, where an additional silane application step was used. In the CS blocks, the CS/AB group (19.32 ± 9.46 MPa) showed significantly greater bond strength than the CS/CO group (10.31 ± 5.90 MPa). Since the structure of CS blocks is more similar to that of polymers, they are classified as resin nanoceramics; these substances consist of a highly cured resin matrix reinforced with approximately 80% by weight nanoceramic particles in combination with various silica nanoparticles, zirconia nanoparticles, and zirconia-silica nanoclusters. Therefore, the higher bond strength values obtained with the AB treatment might be ascribed to the silane-free formula or the compatibility between the adhesive formula and the chemical structure of the block.

The stereomicroscopic analysis of the debonded specimens supported the results of μSBS testing. The VE/AB, VE/CU, and SH/CU groups, which had lower bond strength values, had a higher incidence of adhesive failure (91%, 71%, and 64%, respectively). Only 1 specimen from the VE/CU group showed premature failure. The LA/CO group, which had higher bond strength values than the abovementioned groups, mainly experienced mixed failure (64%). A mixed failure mode is indicative of higher bond strength, while adhesive failure is frequently correlated with lower bond strength [20]. The highest bond strengths were obtained from the CS/AB group, with the following distribution of failure modes: 9% adhesive failure, 55% mixed failure, and 36% cohesive failure. It would be reasonable to expect that the adhesive interface would be the weakest joint. If cohesive fractures were moderately rare, the results would better represent the actual repair strength of the adhesive bond between the block and the new repair composites. More cohesive fractures can be expected as the repair strength approaches the fracture strength of the composite used [16]. We suggest that we obtained bond strength values close to the actual repair values because cohesive resin failure was observed only in a few samples in the study. In all groups, cohesive block failure occurred with considerable frequency, and this result might be related to the thermocycling process. In a study examining the mechanical properties of hybrid CAD/CAM blocks [21], it has been reported that the thermocycling and water storage process adversely affected the hardness and flexural strength of hybrid materials, and the authors speculated that those results were due to water absorption in the matrix resin during the aging process, which caused swelling of the network and reduction in the frictional forces between polymer chains. From the same perspective, the cohesive failure observed in the groups in our study can be attributed to water absorption during two different aging processes.

In the literature, few studies have examined the repair of CAD-CAM hybrid blocks. Ustun *et al.* [14] investigated the SBS of CAD-CAM blocks that were repaired with a Ceramic Repair System and a Clearfil Repair System. No significant difference was found between both repair systems. Similar SBS values were obtained for LA and VE blocks. Elsaka [16] evaluated the repair bond strength of a nanohybrid resin composite applied to a CAD/CAM hybrid ceramic using four intraoral ceramic repair systems. VE blocks repaired with the Cimera zircon and porcelain repair kits showed significantly higher bond strength values than those repaired with the Clearfil repair system and the CoJet system. Higher bond strengths were attributed to the use of hydrofluoric acid and silane agents. The lowest values were obtained from the Clearfil repair treatment, which does not include an additional silane application step. Our findings were in parallel to these results for VE blocks. Stawarczyk *et al.* [11] examined the tensile bond strength of LA blocks that received different surface treatment processes (CoJet air abrasion and Cimara grinder) with different adhesives and repair composites. Groups treated with air abrasion showed statistically significantly greater bond strength. Adhesive and repair composite also affected bond strength, but phosphoric acid treatment and water contamination had no significant effects. The highest bond strength values were obtained from the same manufacturers' block and adhesive system (Lava Ultimate and ScotchBond Universal). Similarly, for the LA groups in our study; the highest bond strength was obtained from the same manufacturer's block and adhesive (LA/SB, 18.17 ± 10.31 MPa), although no significant difference was found in comparison to other adhesive systems.

Although hybrid CAD/CAM blocks vary, the manufacturers recommend similar methods for the repair procedures of these materials in the oral environment: 1) surface roughening (diamond burs or sand-blasting with aluminum oxide), 2) silane or adhesive application, and 3) composite resin application. Universal adhesives, which contain silane or phosphoric acid monomers in addition to regular methacrylic monomers, offer an all-purpose formulation that may enable adhesion to different types of substrates. This has led to the proposal of an important improvement to the bonds of silane or phosphoric monomers, which can prime the inorganic components of the CAD-CAM blocks made of nanoceramic particles embedded in a resin matrix [11,22]. Since this study tested the effects of different universal adhesives, additional silane treatment steps were not necessary. In order to create a standard surface roughening treatment in this study, the CoJet system was applied to all groups [11,15,16,23-26], taking into account its positive effects on bond strength values.

The results of this study showed that all the universal adhesive treatments could be used as an alternative to the control treatment for repair, except for the AB system on VE blocks (VE/AB group); however, the efficiency of each repair system was material-dependent. Previous studies have shown that repair bond strength is influenced by the composition of the repaired substrate, the mechanical and chemical pretreatment processes applied to the surface, the use of silane, the type of adhesives, and the composition of the repair material. Because so many factors affect the success of the repair process, we cannot identify a single ideal repair protocol. In clinical conditions, it is important to know which restorative material will be repaired since that knowledge is important for determining the materials to be used in the repair process. Even though the repair bond strength should be the same as the cohesive strength of the composite resin to re-establish its initial properties, there was no effective way to include 'composite-composite' or 'CAD block-CAD block' control groups [11,26]. Further studies are required to determine the most appropriate repair protocols for hybrid CAD-CAM materials, and different aging conditions should be tested, but laboratory tests cannot be the only reference. Therefore, long-term clinical evaluations of these materials are recommended.

CONCLUSIONS

Within the limitations of this study, μSBS values were affected by the type of hybrid block. All tested universal adhesive treatments can be used as an alternative to the control treatment for repair, except the AB system on VE blocks (VE/AB group). The μSBS values showed variation across different adhesive treatments on different hybrid CAD-CAM block types.

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