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BASIC RESEARCH:

Effect of Different Decontamination Agents on The Bond Strength of CAD/CAM Blocks and Repair Composite Materials

Efecto de diferentes agentes descontaminantes sobre la resistencia de unión de bloques CAD/CAM y resinas compuestas

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ABSTRACT: The purpose of this study was to examine the effect of different decontamination agents on the bond strength of saliva-contaminated CAD/CAM blocks and repair composite materials. Two hunderd eighty 3-mm-thick specimens were prepared from four different CAD/CAM materials: Katana Zirconia UTML disc, IPS e.max CAD block, Shofu block, and Vita Enamic block. Each material was divided into seven different subgroups (N=70). Group 1 had a clean surface. The other groups were comprised of the samples, which were contaminated with human saliva: group 2: negative control (non-cleaned); group 3: cleaned with water spray; group 4: cleaned with 70% ethanol; group 5: cleaned with Ivoclean; group 6: cleaned with Katana Cleaner; and group 7: cleaned with phosphoric acid. After the decontamination protocols, the resin composite cylinders were bonded to the CAD/CAM surfaces with a thin layer of dual-cured resin cement. The samples were stored for 24 hours at 37°C in distilled water. Then, they were subjected to a shear bond strength test (SBS). The values were recorded, and fracture types were evaluated using a microscope. Data were analyzed using two-way ANOVA and Tukey's post-hoc test. Generally, all decontaminating agents improved the SBS of composites to Katana Zirconia UTML, IPS e.max, and Vita Enamic materials ($p<0.05$). However, for Shofu materials, group 6 samples exhibited significantly higher bond strength values as compared with group 2 samples ($p=0.026$). The highest SBS values were seen in the phosphoric acid-treated group in Katana zirconia materials (26.45 ± 9.38 MPa), whereas the lowest values were seen in group 2 samples in Shofu materials (13.17 ±

3.40 MPa). Each decontaminant agent improved the bond strength of composites to the contaminated CAD/CAM materials. If saliva is not cleaned before adhesive procedure, SBS values may decrease. All decontamination agents can be used safely on zirconia, lithium-disilicate glass-ceramic, hybrid ceramic, and polymer-infiltrated ceramic surfaces.

KEYWORDS: Ivoclean; Katana Cleaner; CAD/CAM; Surface decontamination; Shear bond strength; Composite.

RESUMEN: El propósito de este estudio fue examinar el efecto de diferentes agentes descontaminantes sobre la fuerza de unión de bloques CAD/CAM contaminados con saliva y materiales compuestos de reparación. Se prepararon doscientas ochenta muestras de 3 mm de espesor a partir de cuatro materiales CAD/CAM diferentes: disco Katana Zirconia UTML, bloque IPS e.max CAD, bloque Shofu y bloque Vita Enamic. Cada material se dividió en siete subgrupos diferentes (N=70). El grupo 1 tenía una superficie limpia. Los otros grupos estaban compuestos por muestras que estaban contaminadas con saliva humana: grupo 2: control negativo (no limpio); grupo 3: limpiado con agua pulverizada; grupo 4: limpiado con etanol al 70%; grupo 5: limpiado con Ivoclean; grupo 6: limpiado con Katana Cleaner; y grupo 7: limpiado con ácido fosfórico. Después de los protocolos de descontaminación, los cilindros de composite de resina se adhirieron a las superficies CAD/CAM con una fina capa de cemento de resina de curado dual. Las muestras se almacenaron durante 24 horas a 37°C en agua destilada. Luego, fueron sometidos a una prueba de resistencia al corte (SBS). Se registraron los valores y se evaluaron los tipos de fracturas utilizando un microscopio. Los datos se analizaron mediante ANOVA de dos factores y la prueba post-hoc de Tukey. En general, todos los agentes descontaminantes mejoraron el SBS de los composites con los materiales Katana Zirconia UTML, IPS e.max y Vita Enamic ($p<0,05$). Sin embargo, para los materiales Shofu, las muestras del grupo 6 exhibieron valores de fuerza de unión significativamente más altos en comparación con las muestras del grupo 2 ($p=0,026$). Los valores más altos de SBS se observaron en el grupo tratado con ácido fosfórico en materiales de circonio Katana ($26,45 \pm 9,38$ MPa), mientras que los valores más bajos se observaron en las muestras del grupo 2 en materiales Shofu ($13,17 \pm 3,40$ MPa). Cada agente descontaminante mejoró la fuerza de unión de los composites a los materiales CAD/CAM contaminados. Si no se limpia la saliva antes del procedimiento adhesivo, los valores de SBS pueden disminuir. Todos los agentes descontaminantes se pueden utilizar de forma segura en superficies de circonio, cerámica de vidrio de disilicato de litio, cerámica híbrida y cerámica con infiltraciones de polímeros.

PALABRAS CLAVE: Ivoclean; Limpiador de katanas; CAD/CAM; Descontaminación de superficies; Resistencia al corte; Compuesto.

INTRODUCTION

With the advancement of technology, one of the most important goals of restorative dentistry is to restore the integrity of lost dental tissues, function, phonation, and provide aesthetics. It can be said that all-ceramic restorations have an important place in dentistry due to their aesthetic and biocompatible structures (1-3). Today, thanks to computer-aided design and manufacturing (CAD/CAM) technology, restorations can be produced faster, either at the chairside or in the laboratory, and this technology has been used frequently as an alternative to traditional techniques. These systems are one of the fastest-growing and most rapidly changing areas in dentistry (3,4).

In addition to exceptional mechanical and physical qualities, the repairability of dental material is critical for minimally invasive dentistry. Resin-based CAD/CAM materials have been shown to be resistant to functional masticatory forces, but under excessive loads caused by parafunctional habits or trauma, these materials may fracture and the restoration must be completely replaced or repaired (5).

A complete replacement of restorations may cause trauma, substance loss, increased cost, and time loss. However, localized repair of restorations is a less costly, less time-consuming, and more protective treatment option. *In vitro* investigations showed that repaired restorations had higher survival rates than replaced ones (6,7). For these reasons, an intraoral repair can be considered an alternative treatment to changing the restoration for local fractures.

The clinical success of the repair process depends on the preservation of the bonding area (8). Achieving a strong micromechanical bond between the restoration fracture surface and a resin-based composite or resin cement is essential for a successful repair. This connection also involves chemical bonding, thus the selection of appropriate surface treatments for fracture surfaces is necessary (9).

There are abundant data about the effect of several treatment methods on the shear bond strength (SBS) of composite restorations; however, very limited data are available on the use of surface treatment of resin-based CAD/CAM materials to improve their repair bond strength (6-8).

This study has aimed to examine the effects of different decontamination agents on the bond strength of saliva-contaminated CAD/CAM blocks and repair composite materials. The tested null hypothesis was as follows: different decontaminant agents would not affect the SBS of composites to zirconia, lithium disilicate glass-ceramic, hybrid ceramic, or polymer-infiltrated ceramic.

MATERIALS AND METHODS

Two hundred eighty 3-mm-thick samples were prepared from four different CAD/CAM restorative materials: sintered zirconia discs (Katana Zirconia UTML; Kuraray Noritake Dental Inc., Okayama, Japan), lithium disilicate glass-ceramic (IPS e.max CAD; Ivoclar Vivadent, Schaan, Liechtenstein), hybrid ceramic (Shofu; Shofu Dental GmbH, Kyoto, Japan), and polymer infiltrated ceramic (Vita Enamic; Vita Zahnfabrik, Bad Sackingen, Germany). Each material was divided into seven different subgroups (n=10) (Figure 1). The brands, contents, and manufacturers of the tested materials are shown in Table 1.

Seventy samples were obtained from each CAD/CAM material and placed in a self-curing acrylic resin with the surfaces to be tested facing upwards. All samples were wet-grounded with 600-grit silicon carbide (SIC) paper to create standardized surfaces. After that, Katana Zirconia and Shofu samples were sandblasted at 2 bar pressure with 50-µm Al_2O_3 (Renfert GmbH, Hilzingen, Germany) for 10 seconds. IPS e.max samples were etched with hydrofluoric acid (9% Ultradent Porcelain Etch; Ultradent Product Inc.) for 20 seconds, rinsed with water for 10 seconds, and dried with an air syringe for 10 seconds. Vita Enamic samples were etched with hydrofluoric acid for 60 seconds, rinsed with water for 10 seconds, and dried with an air syringe for 10 seconds. Then, all samples were cleaned ultrasonically in distilled water for 15 minutes and dried with an oil-free air syringe.

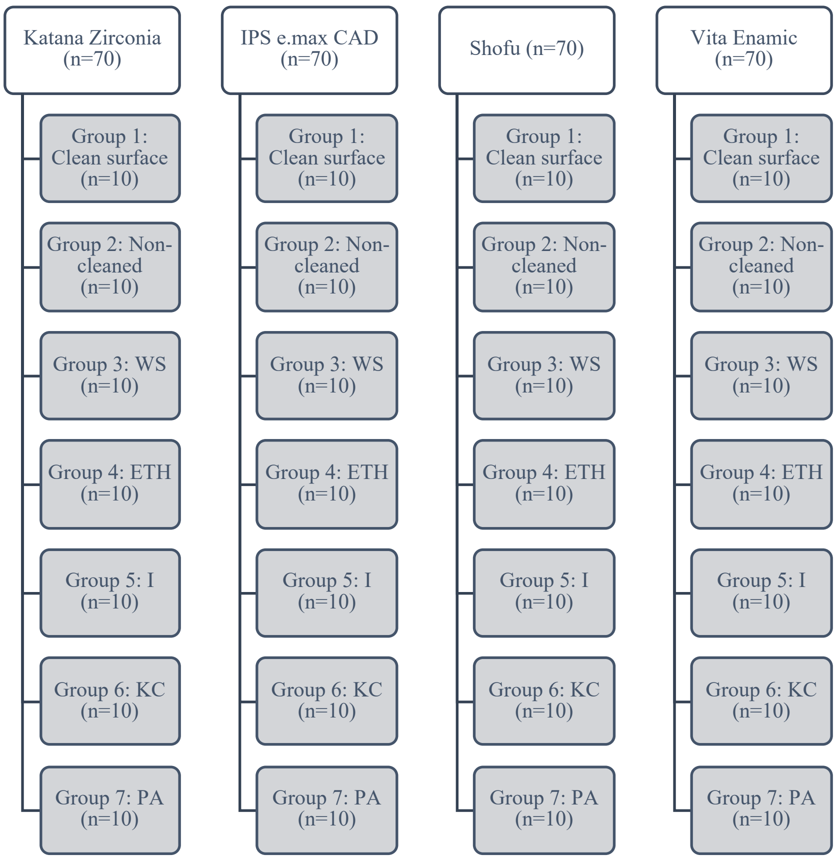

Figure 1. Flowchart of the groups used in the study.

Table 1. The brands, type, contents, and manufacturers of the tested materials.

Material	Type	Contents	Manufacturer
Katana Zirconia UTML	Zirconia disc	ZrO_2, HfO_2, Y_2O_3, pigments	Kuraray Noritake Dental Inc., Okayama, Japan
IPS e.max CAD	Lithium disilicate glass-ceramic	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, other oxides	Ivoclar Vivadent, Schaan, Liechtenstein
Shofu Block	Hybrid-ceramic	UDMA, TEGDMA, SiO_2, zirconium silicate	Shofu Dental GmbH, Kyoto, Japan
Vita Enamic	Polymer-infiltrated ceramic	UDMA, TEGDMA, Feldspar ceramic enriched with aluminum oxide	Vita Zahnfabrik, Bad Sackingen, Germany

ZrO_2: Zirconium oxide, HfO_2: Hafnium oxide, Y_2O_3: Yitrium oxide, SiO_2: Silicon dioxide, Li_2O: Lithium oxide, K_2O: Potassium oxide, P_2O_5: Diphosphorus penta oxide.
ZnO: Zinc oxide, UDMA: Urethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate.

SURFACE TREATMENTS

Each prepared area of the CAD/CAM materials (except group 1) was exposed to 10 microliters of non-stimulated human saliva from one donor for 20 seconds. This experiment was conducted in compliance with the ethical principles of the Declaration of Helsinki and with the permission of the Ethics Committee of Kocaeli University (KOU KAEK 2021/222). The plates were then assigned to seven experimental groups, according to the type of decontamination agent as follows (n=10):

Group 1: Clean surface (no saliva contamination (NS))
Samples of Groups 2-7 were saliva contaminated:
Group 2: Negative control (non-cleaned) (S)
Group 3: Cleaned with water spray (WS)
Group 4: Cleaned with 70% ethanol (ETH)
Group 5: Cleaned with Ivoclean (I)
Group 6: Cleaned with Katana Cleaner (KC) and
Group 7: Cleaned with phosphoric acid (PA)

The full treatment protocol is described in Table 2. Subsequently, a 2.3-mm inner diameter, 3-mm height cylindrical Teflon mold (Ultradent Products Inc., South Jordan, UT) was used to build resin composite cylinders (Clearfil Majesty ES-2, Kuraray Inc., Tokyo, Japan). The composite was placed into the mold and light-cured with a LED curing device (Elipar S10, 3 M ESPE, Germany). After removal from the mold, composite sticks were light-cured on both sides for another 20 seconds.

G-Multi Primer (GC Corporation, Tokyo, Japan) was applied to all surfaces of the CAD/CAM samples. A thin layer of resin cement (G-Cem LinkForce Adhesive Resin Cement; GC Corporation, Tokyo, Japan) was injected onto treated CAD/CAM surfaces. Under a total load of 1 kg, the prepared cylindrical resin composite samples were placed on resin cement and applied to the CAD/CAM surfaces. Samples were light-cured for 5 seconds and excess cement was removed using a dental explorer. All samples were light-cured on both sides for 20 seconds. After 24 hours, the samples were tested using a universal testing device (Bisco Shear Bond Tester, Bisco Inc., Schaumburg, USA). Data were analyzed using two-way analysis of variance (ANOVA) and Tukey's post-hoc test.

FAILURE PATTERN

Following SBS measurements, the failure patterns of the samples were investigated. An optical microscope (M3B, Wild, Heerbrugg, Switzerland) with 50x magnification was used to analyze the interfacial zones. Failure modes were classified as adhesive, cohesive, and mixed failures. A fracture at the composite/ceramic interface was classified as an adhesive fracture (Ad), a fracture within the composite layer or ceramic was classified as a cohesive fracture (Coh), and a mixture of adhesive and cohesive fractures was classified as a mixed fracture (Mix). Fractures were calculated as a percentage of the bonding surface area for each test group.

RESULTS

The SBS results are shown in Table 3. In the comparison of materials in terms of decontamination, there were no statistically significant differences between Katana Zirconia, IPS e.max, and Vita Enamic materials ($p<0.05$). There were significant differences between S and KC groups in Shofu materials ($p=0.026$). When comparing the CAD/CAM materials within themselves, there was a significant difference between Shofu and IPS e.max CAD materials in the S group ($p=0.001$).

The highest SBS value was seen in Katana Zirconia materials in the PA group (26.45 ± 9.38 MPa), and the lowest bond value was seen in Shofu materials in the S group (13.17 ± 3.40 MPa). The failure patterns are shown in Table 4. Most of the Katana Zirconia, IPS e.max CAD, and Shofu samples had adhesive failures, but the Vita Enamic samples had the most cohesion failures.

Table 2. Surface treatment protocols.

Group 1: NS group (Clean surface-no saliva contamination)	A positive control to confirm the initial decontamination of all CAD/CAM materials.
Group 2: S group (Negative control-non-cleaned)	With saliva contamination, and no water wash or decontamination process was considered to be the negative control.
Group 3: WS group	With saliva contamination and washed with sterile water for 10 seconds and no further decontamination afterward.
Group 4: ETH group	With saliva contamination and cleaned with 70% ethanol which apply actively with sterile cotton for 20 seconds and dry with oil-free air.
Group 5: I group	With saliva contamination and applied with a thin layer of Ivoclean with a sterilized microbrush and left on the surface 20 seconds for cleaning action, rinsed with water, and dried with oil-free air.
Group 6: KC group	With saliva contamination and cleaned by Katana Cleaner with a microbrush agitation for 10 seconds, rinsed with water, and dried with oil-free air.
Group 7: PA group	With saliva contamination and cleaned with 37% phosphoric acid (Bisco Inc., Schaumburg, IL, USA) using a micro-brush with agitation for 20 seconds, rinsed with deionized water for 20 seconds, and dried with moisture- and oil-free air.

Table 3. Distribution of mean and standard deviation values according to surface treatment (Mean± standard deviation (MPa)).

	NS	S	WS	ETH	I	KC	PA
Katana Zirconia	24.29±4.58^{Aa}	20.20±7.32^{Aab}	21.51±7.14^{Aa}	24.43±7.90^{Aa}	21.09±6.82^{Aa}	24.72±3.65^{Aa}	26.45±9.38 ^{Aa}
IPS e.max CAD	22.39±6.35^{Aa}	23.26±4.46^{Ab}	19.05±4.44^{Aa}	25.94±4.10 ^{Aa}	22.44±10.01^{Aa}	24.49±6.28^{Aa}	21.28±9.03^{Aa}
Shofu	19.89±9.35^{ABa}	13.17±3.40^{Aa}	19.99±5.37^{ABa}	18.54±6.60^{ABa}	15.23±3.79 ^{ABa}	20.78±4.83^{Ba}	18.56±4.98 ^{ABa}
Vita Enamic	21.40±6.36^{Aa}	19.72± 6.34^{Aab}	23.88±5.26^{Aa}	23.06±8.01^{Aa}	18.76±6.74^{Aa}	23.20±4.70^{Aa}	21.48±6.36^{Aa}

*Different lowercase letters indicate statistically significant differences in the column.
**Different uppercase letters indicate statistically significant differences in the row.

Table 4. Fracture types are shown after shear bond test.

	NS	S	WS	ETH	I	KC	PA
Katana Zirconia	100% Ad (10/10)	100% Ad (10/10)	100% Ad (10/10)	90% Ad (9/10) 10% Coh (1/10)	100% Ad (10/10)	100% Ad (10/10)	90% Ad (9/10) 10% Coh (1/10)
IPS e.max CAD	100% Ad (10/10)	100% Ad (10/10)	100% Ad (10/10)	80% Ad (8/10) 20% Coh (2/10)	100% Ad (10/10)	80% Ad (8/10) 20% Coh (2/10)	100% Ad (10/10)
Shofu	100% Ad (10/10)	100% Ad (10/10)	80% Ad (8/10) 20% Coh (2/10)	100% Ad (10/10)	90% Ad (9/10) 10% Coh (1/10)	70% Ad (7/10) 30% Coh (3/10)	90% Ad (9/10) 10% Coh (1/10)
Vita Enamic	50% Ad (5/10) 50% Coh (5/10)	50% Ad (5/10) 50% Coh (5/10)	10% Ad (1/10) 90% Coh (9/10)	10% Ad (1/10) 90% Coh (9/10)	40% Ad (4/10) 60% Coh (6/10)	10% Ad (1/10) 90% Coh (9/10)	10% Ad (1/10) 90% Coh (9/10)

Ad: Adhesive fracture, Mix: Mixed type fracture, Coh: Cohesive fracture.

DISCUSSION

CAD/CAM restorations used in the clinic are susceptible to fracture due to various reasons such as incorrect adhesive procedures, trauma, or parafunctional habits. If ceramic materials are broken, repair procedures are difficult, and it is a problem in itself for clinicians due to the production technique of ceramics. In addition to being a less-costly technique, the intraoral repair process can also be considered an emergency treatment for localized fractures. Additionally, intraoral repair techniques can be accepted as a minimally invasive because the restoration is difficult to remove, it can damage the tooth during removal, and also the replacement of the restoration is expensive (10-12).

Concerns have increased regarding fracture resistance, durability, and clinical longevity of adhesively cemented restorations made from CAD/CAM blocks (13). For this reason, intraoral repair of fractures encountered in CAD/CAM materials was determined as the subject of this study. In our study, different decontamination agents or protocols were applied to Katana Zirconia, IPS e.max CAD, Shofu, and Vita Enamic block samples. Their bond strengths with resin composite and failure types specific to each group were examined.

Intraoral ceramic repair is performed by replacing the missing piece with resin composite after the surface protocols are applied. The strength and integrity of the bonding between the restoration and the resin composite affect the clinical success of the repair (8). To provide adhesion to dental ceramics, the first step is to increase the surface roughness using surface protocols. Increased surface roughness increases wettability by lowering the surface tension and creating micromechanical retention (14,15).

In our study, the decontamination process with water spray, 70% ethanol, Ivoclean, Katana cleaner, and phosphoric acid was compared on different surfaces (CAD/CAM materials) contaminated with human saliva.

One of the basic principles of bonding is that the bonded area surfaces must be clean. Manufacturers clearly state the limits of bonding protocols. However, it may not always be possible to provide these ideal conditions in the oral environment. For this reason, saliva contamination on adherent surfaces, which often occurs, must be cleaned to ensure the ideal connection. After saliva contamination, the bond strengths of traditional materials such as zirconia, ceramics, and metals are significantly reduced (16-18). When the surface of the restoration is contaminated with saliva, salivary proteins adhere to the restoration's surface through non-covalent adsorption, generating an organic coating that cannot be entirely removed by water cleaning. In our study, it was observed that the bond strength (SBS) values of the samples cleaned with water spray (WS) increased, but not as much as the group that was not contaminated with saliva (NS).

Ethanol (70%) appears to disrupt membranes and quickly denature proteins, interfering with metabolism and leading to cell lysis (19). Ivoclean has been reported to be more effective in cleaning saliva contamination. After ethanol and Ivoclean application, there was an increase in SBS values compared with the S group. Ivoclean can be used for many materials. However, its use in the oral cavity is contraindicated due to its high alkalinity (pH 13.5) (20,21). Accordingly, it can only be used in the extraoral cavity and with de-cemented materials.

Katana Cleaner, which can be used for a wide range of applications, dental ceramics, resin materials, dental posts, dental metal enamel, and dentin, is weakly acidic (pH 4.5), and it can also be used in the oral cavity. It contains MDP salt (surfactant) as an active substance. There were significant differences between the SBS values of Shofu materials and other CAD/CAM materials. The filler amount of Shofu blocks may be the reason for these differences.

In this study, the highest SBS values were observed in Katana Zirconia, in which the surfaces were cleaned with 40% phosphoric acid. Phosphoric acid is commonly found in dental offices. It is an acid-based material that removes organic contaminants and provides a clean surface for successful binding (22, 23). However, different opinions have been expressed about the effectiveness of the use of phosphoric acid in cleaning contaminated surfaces (24). The cleaning of zirconia surfaces with alcohol before phosphoric acid may cause oxide binding, which increases the chemical bonding with phosphate monomers in self-etch adhesive systems and cement. Therefore our null hypothesis, ‘different decontaminant agents would not affect the shear bond strength of composites to zirconia, lithium disilicate glass-ceramic, hybrid ceramic, or polymer-infiltrated ceramic,’ was rejected. On the other hand, the researchers found no differences between the different tested cleaning protocols (25).

The examination of the binding success is related to both shear test results and the type of fracture. Atsu *et al*. stated that cohesive and mixed (adhesive+cohesive release) release types indicated a higher SBS than the adhesive type, and the adhesive type was associated with low bond strength (26). However, most failure patterns were found to be adhesive in our study. Further studies are needed to understand these features.

CONCLUSION

Within the limitations of this study, each decontaminant agent improved the bond strength of composites to saliva-contaminated CAD/CAM materials. If saliva is not cleaned before the adhesive procedure, SBS values may decrease significantly for some materials. For this reason, the success of surface treatments may vary according to the CAD/CAM material type. Further studies are required for the optimal surface treatment.

CONFLICT OF INTEREST

The authors declare no competing interests.

ETHICAL APPROVAL

This study was approved by the Ethics Committee of Kocaeli University (KOU KAEK 2021/222).

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AUTHOR CONTRIBUTION STATEMENT

Conceptualization and design: D.K. and N.T.
Literature review: D.K. and E.İ.S.
Methodology and validation: D.K. and N.T.
Formal analysis: E.İ.S.
Investigation and data collection: D.K.
Resources: D.K. and E.İ.S.
Data analysis and interpretation: D.K
Writing-review and editing: D.K. and N.T.
Supervision: N.T.
Project administration: D.K. and N.T.

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