Brazilian Dental Science

unesp
UNIVERSIDADE ESTADUAL PAULISTA "JÚLIO DE MESQUITA FILHO"
Instituto de Ciência e Tecnologia
Campus de São José dos Campos

ORIGINAL ARTICLE

DOI: https://doi.org/10.4322/bds.2023.e3978

Effect of adhesive primer application on shear bond strength of self-adhesive cement to tooth structure and two different CAD/CAM milled blocks

Efeito da aplicação do primer adesivo na resistência ao cisalhamento do cimento autoadesivo à estrutura dentária e a dois diferentes blocos fresados CAD/CAM

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How to cite: Issa MI, Abdulazeez MI, Fatalla AA. Effect of adhesive primer application on shear bond strength of self-adhesive cement to tooth structure and two different CAD/CAM milled blocks. Braz Dent Sci. 2023;26(4):e3978. https://doi.org/10.4322/bds.2023.e3978

ABSTRACT

Objective: This study was conducted to evaluate the influence of the G-CEM ONE adhesive enhancing primer on the shear bond strength of self-adhesive resin cement (G-CEM ONE) to both tooth structure and two different CAD/CAM blocks (GC Initial LiSi and Cerasmart 270). **Material and Methods:** Forty specimens (cylindrical-shaped, 5 mm in diameter and height) were milled from both CAD/CAM blocks (20 specimens from each block type). Forty sound upper premolars were sectioned to the level of peripheral dentin, then randomly divided into four groups (n = 10): A1: GC Initial LiSi without adhesive enhancing primer; B1: Cerasmart 270 without adhesive enhancing primer; A2: GC Initial LiSi with adhesive enhancing primer; B2: Cerasmart 270 group with adhesive enhancing primer application. The CAD/CAM blocks were cemented on teeth using a self-adhesive resin cement (G-cem one). The shear bond strength was assessed using a computerized universal testing machine. A digital microscope was used to study the mode of failure. The shear bond strength values data were analyzed statistically using paired *t*-test and independent *t*-test at the significance level of (0.05). **Results:** A significant difference was shown in the shear bond strength values among groups ($P = 0.000$). The highest shear bond strength value was revealed in group A2, while group B1 exhibited the lowest shear bond strength value. **Conclusion:** Using the adhesive enhancing primer on the tooth's surface improved the resin cement's bond strength to CAD/CAM blocks. Additionally, GC Initial LiSi exhibited higher bond strength than Cerasmart 270, with or without the primer.

KEYWORDS

Adhesive; CAD/CAM; Dentin; Resin cement; Shear strength.

RESUMO

Objetivo: Este estudo foi conduzido para avaliar a influência do primer adesivo G-CEM ONE na resistência ao cisalhamento do cimento resinoso autoadesivo (G-CEM ONE) tanto na estrutura dentária quanto em dois diferentes blocos CAD/CAM (GC Initial LiSi e Cerasmart 270). **Material e Métodos:** Quarenta corpos de prova (formato cilíndrico, 5 mm de diâmetro e altura) foram fresados em blocos CAD/CAM (20 corpos de prova de cada tipo de bloco). Quarenta pré-molares superiores sadios foram seccionados até o nível da dentina mais externa e, em seguida, divididos aleatoriamente em quatro grupos (n = 10): A1: GC Initial LiSi sem primer adesivo; B1: Cerasmart 270 sem primer adesivo; A2: GC Initial LiSi com primer adesivo; B2: Grupo Cerasmart 270 com aplicação de primer adesivo. Os blocos CAD/CAM foram cimentados nos dentes com cimento resinoso autoadesivo (G-CEM ONE). A resistência ao cisalhamento foi avaliada utilizando uma máquina de ensaios universal computadorizada. Um microscópio digital foi utilizado para estudar o modo de falha. Os dados dos valores de resistência ao cisalhamento foram analisados estatisticamente por meio do teste t pareado e teste t independente ao nível de significância de (0,05). **Resultados:** Foi demonstrada diferença significativa nos valores de resistência ao cisalhamento entre os grupos (P =0,000). O maior valor de resistência ao cisalhamento foi revelado no grupo A2, enquanto o grupo B1 exibiu o menor valor de resistência ao cisalhamento. **Conclusão:** A utilização do primer adesivo na superfície dentária melhorou a resistência de união do cimento resinoso aos blocos CAD/CAM. Além disso, o GC Initial LiSi apresentou maior resistência de união que o Cerasmart 270, com ou sem primer.

PALAVRAS-CHAVE

Adesivo; CAD/CAM; Dentina; Cimento resinoso; Força de cisalhamento.

INTRODUCTION

Recent advances in dental technologies have increased the popularity of different computer-aided design /computer-aided manufacturing (CAD/CAM)-machinable materials, including glass ceramics, resin-based Composites, and polymer-infiltrated ceramics [1]. Among these materials, ceramics have the advantage of natural-looking, wear resistance, durability, and biocompatibility [2,3]. Yet, ceramic restorations could be more susceptible to chipping due to their brittle nature [4]. Lithium disilicate (LiSi) glass-ceramic is one of the most widely used ceramic systems in aesthetic dentistry due to its high optical and mechanical properties. GC Initial LiSi is a fully crystallized lithium disilicate CAD/CAM ceramic with the advantage of decreasing the chair side time as it does not require further heat treatment after milling [5,6].

Although composite resin blocks have lower flexural strength than ceramics, they have the advantages of low abrasiveness to the antagonist, ease of modification and repair, reduced milling time, and less wear of milling tools [7,8]. CAD/CAM composite resin matrix ceramics were developed to combine the advantages of composites and ceramics [9,10]. These blocks can be classified into two types depending on their microstructure: materials with a polymer-infiltrated ceramic network (PICN) such as VITA Enamic and materials with dispersed fillers; nanohybrid-composite with inorganic ceramic fillers such as Cerasmart and Lava Ultimate [11]. PICN materials consist of a glass/ceramic network, frequently pre-sintered, that has been silanated by capillary action and then infiltrated with a resin matrix, while materials with dispersed fillers consist of a methacrylate-based matrix similar to that of direct composite materials reinforced by different types and sizes of silanated fillers [12]. Cerasmart is a resin nano-ceramic block containing composite resin material (BisMEPP, UDMA, DMA) with 71% silica and barium glass nanoparticles by weight [13].

Durable bonding between the cement and restorative material is crucial for the longevity of the indirect restoration. Adhesive luting of the restoration has two bonding interfaces; thus, the type of resin cement significantly impacts the restoration's clinical outcome [14]. Introducing self-adhesive resin cement could simplify the multi-steps of adhesive bonding and minimize the possibility of handling errors and post-operative sensitivity [15]. This type of cement provides micromechanical retention due to the presence of acidic monomers that could demineralize and penetrate the tooth surface. Additionally, the reaction between the acidic monomers of the cement and hydroxyapatite of the tooth substrate results in chemical retention. Yet, some studies have stated that self-adhesive resin cement provides poor adhesion to dentine that could reduce the bond strength [16,17].

A new self-adhesive resin cement called G-CEM ONE (GC Corporation, Tokyo, Japan) was recently introduced with an additional agent, G-CEM ONE Adhesive enhancing primer) GC Corporation, Tokyo, Japan) [18]. This primer is comprised of acidic monomers such as 4-2-(methacryloyloxy)ethoxy] carbonyl phthalic acid (4-MET) and 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) that could enhance the infiltration of the cement in the dentinal tubules. Moreover, the "touch-curing catalyst" in the primer is claimed to enhance the chemical polymerization of the cement [19,20].

Limited information is available in the literature concerning this adhesive primer; thus, the present study was applied to investigate the influence of G-CEM ONE adhesive enhancing primer on the shear bond strength (SBS) of self-adhesive resin cement; G-CEM ONE to both tooth structure and two different CAD/CAM blocks; GC Initial LiSi [lithium disilicate] and Cerasmart 270, and evaluating the mode of failure. The first null hypothesis is the application of the adhesive enhancing primer doesn't affect the shear bond strength between the tooth structure and CAD/CAM blocks. The second null hypothesis, there is no difference in the shear bond strength of different CAD/CAM blocks to tooth structure using self-adhesive resin cement.

MATERIALS AND METHODS

Preparation of CAD/CAM specimens

Forty cylindrical-shaped specimens (5 mm in diameter and height) were milled from both CAD/CAM blocks using an In-Lab MC X5 milling device (Sirona, Germany). 20 specimens from GC Initial LiSi (GC Corporation, Tokyo, Japan) and 20 specimens from Cerasmart 270 (GC Corporation, Tokyo, Japan). The bonding surface of each cylinder was then treated with silicon carbide abrasive paper #grit 80 under water cooling using a grinding and polishing machine (MOpao 160E, China) to provide standardized roughness [21]. The specimens were then ultrasonically cleaned in an ultrasonic unit with distilled water for 5 minutes to remove contaminants.

Preparation of teeth specimens

This work has been approved by the research ethics committee (ref. number 808, project number 808223 at 18.5.2023). Forty sound human upper 1st premolars extracted for orthodontic purposes were collected. The selected samples were intact and free from caries, cracks, and restorations. The teeth were washed with distilled water and stored in normal saline 0.9% at room temperature. Each tooth specimen was then embedded in acrylic, which was used as a holding block. A line 3 mm occlusal (above) the cemento-enamel junction was drawn on each tooth to determine the area to be embedded in acrylic. A custom-made silicon mold with dimensions (1.5× 1.5× 2 cm) was used to aid in the construction of standardized acrylic holding blocks, this mold acted as a container to hold the acrylic during the setting time. Each tooth was then attached to the dental surveyor and embedded along its long axis in cold cure acrylic (Veracril, Colombia) till the drawn line and the acrylic allowed to set. The occlusal surface of each tooth was reduced down to the level of the central groove to expose the peripheral dentine surface. Tooth reduction was done using a diamond disc in a straight handpiece mounted to a dental surveyor with water cooling. The cut surface of each tooth was then finished with silicon carbide abrasive papers grit #220, then grit # 500 with water cooling using the same grinding and polishing machine that was used for finishing the CAD/CAM specimens to provide a standardized surface roughness of all specimens [21]. The prepared tooth and CAD/CAM specimens are shown in Figure 1.

Sample grouping

The prepared samples were assigned into two groups (20 teeth for each group) according to the type of the cemented CAD/CAM block. Each group was subdivided into two subgroups (10 teeth for each subgroup) according to the application or not of the adhesive primer:

Group A1: GC Initial LiSi group without adhesive enhancing primer application.

Group B1: Cerasmart 270 group without adhesive enhancing primer application.

Group A2: GC Initial LiSi group with adhesive enhancing primer application.

Group B2: Cerasmart 270 group with adhesive enhancing primer application.

The list of the main materials used in this study is shown in Table I.

Cementation procedure

Bonded surface treatment of CAD/CAM blocks

The bonded surface of each block was conditioned and treated following the

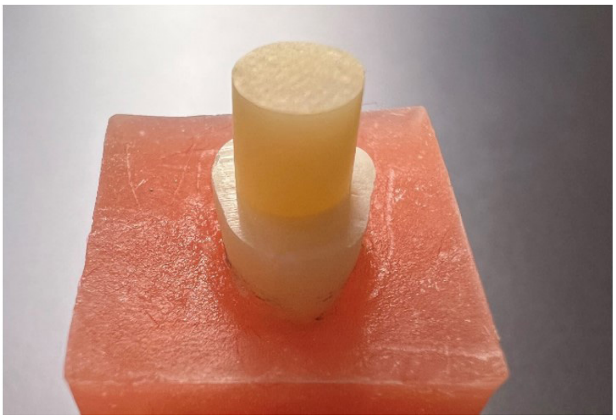

Figure 1 - The prepared tooth and CAD/CAM specimen.

Table I - Chemical composition and manufacturer of materials used

Material	Manufacturer	Chemical composition (wt%)
GC Initial ™ LiSi Block	GC Corporation, Tokyo, Japan LOT 2112081	SiO_2: 81%, P_2O_5: 8.1%, K_2O: 5.9%, Al_2O_3: 3.8%, TiO_2: 0.5%, CeO_2: 0.6%
CERASMART™ 270 Block	GC Corporation, Tokyo, Japan LOT 2202071	Bis-MEPP, UDMA, DMA, Silica (20 nm), barium glass (300 nm) 71 wt%
G-CEM One self-adhesive cement	GC Corporation, Tokyo, Japan LOT 2206061	Paste A: fluoroaluminosilicate glass, UDMA, dimethacrylate, initiator, stabilizer, pigment, silicon dioxide, MDP, Paste B: SiO_2, trimethoxysilane, MgO, UDMA, 2-hydroxy-1,3 dimethacryloxypropane, MDP, 6-tert-butyl-2,4-xylenol, 2,6-di-tert-butyl-p-cresol, EDTA disodium salt dehydrate, vanadyl acetylacetonate, TPO, ascorbic acid, camphorquinone,
G-CEM One adhesive enhancing primer	GC Corporation, Tokyo, Japan LOT 2206061	Ethanol, MDP, 10-methacryloyloxydecyl dihydrogenthiophosphate, 4-META, 2-hydroxy-1,3-dimethoxypropane, vanadyl acetylacetonate, 2,6-di-tert-butyl-p-cresol
G-Multi Prime	GC Corporation, Tokyo, Japan LOT1611181	MDP, MDTP, γ-MPTS, methacrylate, monomer, ethanol

manufacturer's recommendations. For all groups, the bonded surface was conditioned with hydrofluoric acid 5% for 20 seconds in groups (A1&A2) and 60 seconds in groups (B1&B2), then washed and dried. After that, a silane coupling agent (G- multiprimer) was applied for 60 seconds and then air dried.

Surface treatment of the teeth

The bonded surfaces of the teeth in Groups (A1 and B1) were left without any treatment, while for Group (A2 and B2) the G-cem one adhesive enhancing primer was applied to the bonded interface of each tooth and left undisturbed for 10 seconds after the end of the application and then dried thoroughly for 5 seconds under maximum air pressure.

Cementation with the resin cement

The self-adhesive resin cement (G-cem one) was applied on the bonded surface of each CAD/CAM specimen which was then seated on the tooth under a constant load of 5 Kg with the aid of a dental surveyor, the excess cement was removed, and each surface was light cured for 20 seconds with Curing Pen light cure device (Eighteeth, Changzhou, China) (Light intensity:1000 Mw/cm^2). The specimen was then removed from the cementation device and stored in distilled water for 24 hours [22].

Assessment of shear bond strength

A computer-controlled universal testing machine (Laryee Tchnology co.,Ltd., Model: DWD-50,China) was used to assess the shear bond strength. The shear force was applied on the adhesive interface of each specimen using a knife-edge chisel rod at a crosshead speed of 1 mm/min and 50 KN load cell, as shown in Figure 2. Then, the maximum failure load was recorded in Newton (N) when the fracture occurred. The shear bond strength values in MPa were calculated by dividing the failure load (N) by the bonding area (mm^2).

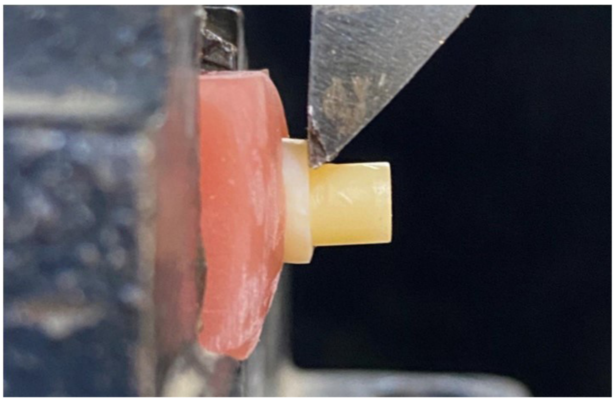

Figure 2 - Test specimen in the computer-controlled testing machine.

$$Shear\ bond\ strength(SBS)\ value = failure\ load\ value(N)/area(mm^2) \quad (1)$$

Statistical analysis

Statistical Package for Social Science (SPSS) was used to analyze the collected data, and the normality of the distribution of variables was assessed using the Shapiro–Wilk test. Paired *t*-test was used to study the effect of the adhesive primer on the shear bond strength. An Independent *t*-test was used to assess the differences in the shear bond strength values between the different CAD/CAM blocks.

Failure mode analysis

To study the mode of Failure, A Dino-Lite digital microscope at a magnification of 50x was used to examine the fractured specimens after debonding.

The Failure modes were classified as follows [23]:

1. Cohesive failure in dentin (CD).
2. Adhesive failure between the dentin and resin cement (ADR).
3. Cohesive failure in the adhesive resin cement (CR).
4. Adhesive failure between the resin cement and the CAD/CAM block material (ARB).
5. Cohesive failure in the CAD/CAM block material (CB).

RESULTS

The descriptive statistics of the data, including the mean and standard deviation of the shear bond strength values in MPa for all groups, are shown in Figure 3. The samples in group A2 (GC Initial LiSi with adhesive enhancing primer) recorded the highest mean value of the shear bond strength (19.082± 2.018 MPa), whereas the lowest mean value (7.490± 1.277 MPa) was recorded by group B1 (Cerasmart 270 without adhesive enhancing primer).

Independent t-test was used to compare the shear bond strength values between the groups with and without the application of the primer at a level of significance of 0.05 and showed a statistically significant difference among the groups (*P*= 0.000; Table II). Independent *t*-test test revealed a statistically significant difference in the shear bond strength values between the different CAD/CAM blocks (Table III). The failure modes distribution in percentages in each group is shown in (Table IV and Figure 4).

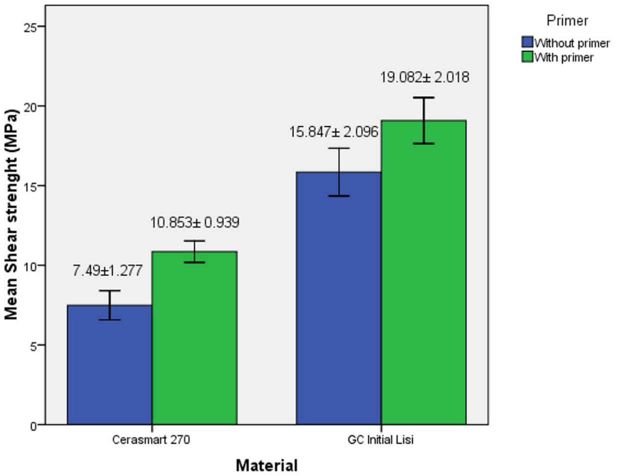

Figure 3 - Bar chart shows the mean values of shear bond strength ± standard deviation.

DISCUSSION

The bonding performance of self-adhesive resin cement remains an issue that is critical for the longevity of the restoration. Previous studies showed that the surface treatment of the dentin could enhance the bond strength of the self-adhesive resin cement [19,24-28]. Based on the results of the present study, the treatment of the tooth surface with the adhesive enhancing primer could significantly improve the shear bond strength of the self-adhesive resin cement. Therefore, the first null hypothesis was rejected.

The most reliable adhesive strategy for the cementation of indirect restoration is still debatable. The self-adhesive resin cement is based on the presence of functional monomers that

Table II - Independent t-test comparing the shear bond strength values between the groups with and without primer applications

Group	Mean ± SD	*t*	*P* value
A1	15.847± 2.096	- 6.705	0.000
A2	19.082± 2.018		
B1	7.490± 1.277	- 3.516	0.002
B2	10.853± 0.939		

SD: Standard deviation.

Table III - Independent t-test comparing the shear bond strength values between different CAD/CAM blocks groups

Group	Mean ± SD	*t*	*P* value
A1	15.847± 2.096	10.726	0.000
B1	7.490± 1.277		
A2	19.082± 2.018	11.688	0.000
B2	10.853± 0.939		

SD: Standard deviation.

Table IV - Mode of failure distribution in each group in (%)

Group	CD	ADR	CR	ARB	CB
A1	0	80%	20%	0	0
A2	60%	0	40%	0	0
B1	0	80%	20%	0	0
B2	20%	40%	40%	0	0

CD: Cohesive failure in dentin; ADR: Adhesive failure between dentin and resin cement; CR: Cohesive failure in resin cement; ARB: Adhesive failure between resin cement and CAD/CAM block material; CB: Cohesive failure in CAD/CAM block material.

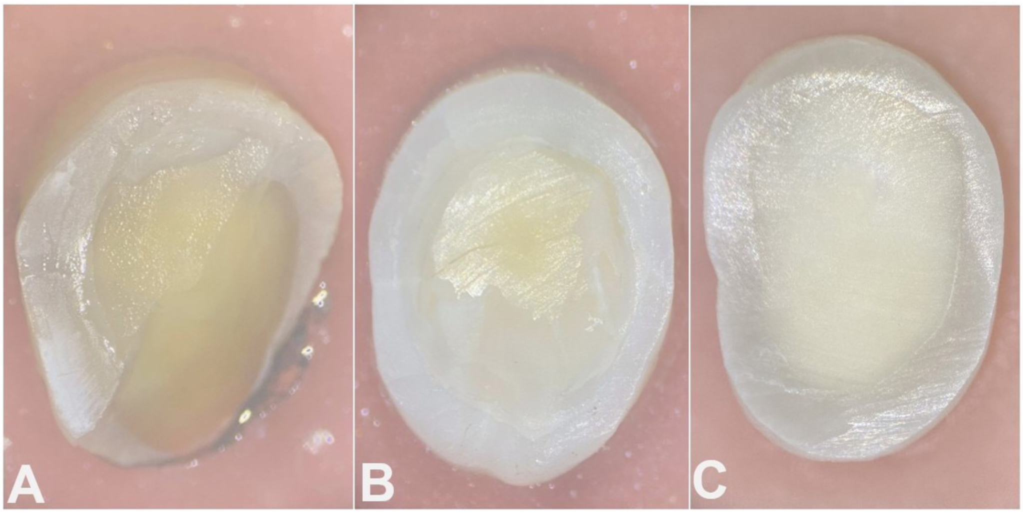

Figure 4 - Modes of failure after shear bond strength test: (A) Cohesive failure in dentin; (B) Cohesive failure in resin cement; (C) Adhesive failure between dentin and resin cement.

could improve the chemical and micromechanical retention, minimize the multi-steps of cementation, and reduce the post-operative sensitivity [29,30]. Yet, these monomers have a lower PH than phosphoric acid, resulting in a limited capacity for demineralization and incomplete removal of the smear layer. Moreover, a study observed that no resin tag or true hybrid layer was formed when the self-adhesive cement was used [27].

The factors mentioned above could be responsible for the lower shear bond strength values presented in the current study for both GC Initial LiSi and Cerasmart 270 groups when the self-adhesive resin cement was used without the adhesive enhancing primer.

The application of adhesive enhancing primer with the self-adhesive resin cement showed a significant increase in the shear bond strength values. This could be explained by the functional monomers in the primer as methacryloyloxydecyl dihydrogen phosphate and 4-2-(methacryloyloxy)ethoxy]carbonyl phthalic acid (4-MET). 10-MDP could link with hydroxyapatite to form MDP–Ca salt that could protect the exposed collagen [31]. Likewise, MDP has a hydrophobic part that plays a significant role in the preservation of collagen, which seems imperative for dentin bonding [32]. On the other hand, 4-MET monomer has hydrophilicity and could enhance the wetting and flowability of the primer [33]. Therefore, both monomers could increase the resin infiltration into the dentine, allowing the self-adhesive resin cement to create a thicker hybrid layer and a stronger bond with the tooth.

Another contributing factor to the high bond strength obtained with the primer is the “touch-curing catalyst” that provides the self-polymerization of the G-CEM cement when it comes in contact with the primer; therefore, the water absorption of the cement was decreased, and this might overcome the negative impact of the moisture which is critical for bonding to the dentin [34]. Additionally, it has been shown that applying the adhesive enhancing primer could increase the free radical reactions, resulting in a higher degree of conversion and enhanced mechanical properties of the self-adhesive resin cement [35].

The results presented in this study are consistent with previous studies [18-20] that all showed an improvement in the shear bond strength of self-adhesive resin cement after treating the tooth surface with G-CEM ONE Adhesive enhancing primer. The authors recorded the highest bond strength with this primer compared to the other materials tested in their studies (polyacrylic acid, self-etch bond, NaOCl).

Likewise, Atalay et al. [22] pointed out that the application of the adhesive enhancing primer before G-CEM resin cement could increase the SBS to both enamel and dentin. The authors supported their findings with SEM examination that showed a more regular pattern of the bonded interface with the use of the primer.

The current study revealed a statistically significant increase in the shear bond strength of the self-adhesive resin cement to the GC Initial LiSi with and without the primer as compared to the Cerasmart 270; therefore, the second null hypothesis was rejected. The different chemical compositions and microstructure of the tested CAD/CAM blocks and the type of surface treatment performed on these materials could be mainly responsible for this finding.

Different Chemical and mechanical methods of surface treatment have been indicated to clean the bonded surface, increase the surface energy and wettability, and consequently improve the adhesive bond between the restoration and resin cement [36-38]. For lithium di-silicate ceramics, HF etching seems to be the gold standard method for surface treatment. The glassy phase of ceramics dissolved with etching, leaving the crystalline phase more visible. This was supported by previous studies, which demonstrated an improvement in the bond strength of different CAD/CAM ceramics when conditioned with HF acid before bonding [39-42].

Both sandblasting and HF etch are indicated for nano-ceramics. In the current study, the bonded surface of GC Initial LiSi and Cerasmart 270 was treated with 5% HF acid following the manufacturers' instructions. Thus, the highest shear bond strength values obtained with GC Initial LiSi could be attributed to their highly glassy content as compared with Cerasmart 270, the glassy phase dissolved HF acid, resulting in the formation of micro retention that increased the surface energy and improved the bond between the cement and the restoration [43]. The finding of this study is in line with other studies, which demonstrated higher SBS values with the lithium di-silicate than with Cerasmart after conditioning with HF [43,44].

It has been shown that air abrasion seems to be the best surface treatment method for indirect composite restorations. Studies revealed that sandblasting resulted in higher surface roughness than HF acid, leading to an increase in the bonded surface area and, in turn enhance the shear bond strength [45-47]. However, the opposite was reported by other studies [43,48]. These contradicting results might be explained by the different types of resin cement and CAD/CAM blocks tested by the authors.

Regarding the mode of failure, the results of the study presented a higher percentage of adhesive failure between dentin and resin cement in groups without adhesive enhancing primer application (A1, B1). This could be attributed to the self-adhesive cement that cannot completely remove the smear layer, which can cause a weak hybrid layer between the cement and the dentin [49]. On the other hand, for groups with the adhesive enhancing primer application (A2, B2), most of the specimen failure was due to cohesive failure in dentin or cohesive failure in resin cement, and this complies with the results of the SBS test that both support the positive effect of the primer on the bond between the dentin and the cement. This finding showed agreement with a study revealed that cohesive failure was the most encountered failure when the adhesive enhancing primer was used [20].

Different methods have been advocated to evaluate the effectiveness of the interfacial adhesive bond of the restorations and adhesive materials to the tooth; among these methods, the shear bond strength test was used due to its simplicity and low- technique sensitivity [50]. Moreover, the Macro shear test is performed without the need for sectioning procedures to obtain specimens, which may induce early micro-cracking [51]. On the other hand, the Micro shear tests have the advantage of smaller bonding areas that could overcome concerns regarding the heterogenous stress pattern of Macro shear tests [50]. Yet, there is no standardized recommended protocol for bond strength assessment [52].

One of the limitations of this study was that it was conducted in *vitro*; thus, it was difficult to imitate the oral cavity environment. Another limitation is that the adhesive enhancing primer was tested with one type of self-adhesive cement. Moreover, the SBS was measured after a short time with no ageing, which could be more clinically relevant.

CONCLUSIONS

Within the limitation of this study, the following conclusions could be drawn:

1. The surface treatment of the tooth with the adhesive enhancing primer improved the shear bond strength of the self-adhesive resin cement to the tooth and both types of CAD/CAM blocks;
2. The shear bond strength of the self-adhesive resin cement to the GC Initial LiSi with and without the primer was higher than that of the Cerasmart 270.

Acknowledgements

The authors would like to thank GC Corporation, Tokyo, Japan and their dealer Vital Allience Co Ltd. for their assistance and support.

Author's Contributions

MII: Conceptualization, Methodology, Resources, Data Curation, Writing – Review & Editing. MIA: Investigation, Writing – Review & Editing, Visualization. AAF: Conceptualization, Methodology, Writing – Review & Editing.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the research ethics committee of the college of dentistry, university of Baghdad. The approval code for this study is (ref. number 808, project number 808223 at 18.5.2023).

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Date submitted: 2023 Aug 02
Accept submission: 2023 Sept 13