

BOND DURABILITY OF DIFFERENT RESIN CEMENTS TO NANOHYBRID CAD/CAM CERAMIC MATERIAL

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ABSTRACT

Aim: This study was conducted to evaluate the effect of self etch adhesive based resin cement and self adhesive resin cements on the shear bond strength SBS of CAD/CAM nanohybrid ceramics bonded to dentin with and without thermocycling.

Materials & methods: Forty molar teeth were selected. The buccal enamel was removed to expose dentin. The crowns were embedded in autopolymerizing acrylic resin. Forty circular specimens of 2.5 mm thickness and 2.5 mm diameter were obtained from CAD/CAM nanohybrid ceramic (Shofu Disc HC) using a low-speed diamond saw with coolant. Ceramics samples were divided into two groups of 20 discs each according to the cement applied either self-etch or self-adhering resin cements. Half of the samples in each group (10 samples) were subjected to thermocycling. Each specimen was thermally aged in a 5°C to 55°C water bath for 5000 cycles. The shear bond strength tests SBS were carried out using a universal testing machine. Failure modes were observed under stereomicroscope.

Results: Self-etch resin cement showed higher statistically significant SBS values than self adhesive cements. Thermocycling showed a statistically significant lower SBS values in all the groups.

Conclusion: Within the limitations of this study, it could be concluded that, Self-etch adhesive based resin cement is preferred over self-adhering resin cement in bonding of Shofu Disk HC nanohybrid CAD/CAM ceramic to dentin. Thermocycling showed detrimental effect on SBS of Shofu Disk HC nanohybrid CAD/CAM ceramic to dentin regardless the cement type.

KEYWORDS: self-etch, self adhesive, resin cement, nanohybrid CAD/CAM ceramic, shear bond strength

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INTRODUCTION

The improvement of computer-aided design and computer-aided manufacturing (CAD/CAM) technology had a great influence on prosthodontics and restorative dentistry [1]. CAD/CAM technology has the advantage of the simplicity of clinical procedures, reduced time, and cost of fabrication of indirect restoration [2]. CAD/CAM nano-ceramic materials combine the simple use as resin composite, with the good mechanical properties and finishing of ceramics [3]. The manufacturers developed resin-matrix ceramic materials to achieve a material with modulus of elasticity closer to dentin, and easier to mill than glass-matrix or polycrystalline ceramics. [4].

The latest version of resin– matrix ceramics launched by shofu is called hybrid resin nanoceramic. It is composed of 61% zirconium silicate placed in a nanofiller composite which forms a skeleton that can absorb masticatory forces and enhance fracture resistance. In comparison to other CAD/CAM materials, Shofu Block & Disk HC showed better milling time, fracture resistance, and can be fabricated in low thickness [5].

Adhesion between the ceramic restoration and the tooth is essential for longevity. Strong bonding through adhesive resin cementation increases fracture strength and allows mechanical integration of the system. Also, strong adhesion enhances adaptation and decreases marginal leakage and recurrent caries [6].

Two types of resin cements were developed, adhesive-based cements which require the use of an adhesive, and self-adhesive cements which do not require any pretreatment. Adhesive systems used with adhesive-based cements are classified into total etch (TE) and self-etch (SE) [7]. Both systems seek one or two steps before application. On contrary, self-adhesive resin cements require less time and number of steps during bonding procedures [7,8]. Self-adhesive cements include in

their structure phosphate groups that can react with the hydroxyapatite and the ceramic surface. In the literature, self-adhesive resin cements showed better results when bonded to aluminum-oxide, leucite-reinforced, and lithium disilicate ceramics [9]. However, further research is required to test the effect of the new available cements on the bond strength of recent CAD/CAM materials.

Therefore, the aim of this study was to evaluate the effect of selfetch adhesive based resin cement and selfadhesive resin cements on the shear bond strength SBS of CAD/CAM nanohybrid ceramics bonded to dentin with and without thermocycling. The null hypotheses were that:

- (1) The adhesive resin cement type will not affect the SBS between the CAD/CAM nanohybrid ceramic material and the dentin,
- (2) Thermocycling has no effect on SBS of CAD/CAM nanohybrid ceramic bonded to dentin.

MATERIALS AND METHOD

Materials' manufacturer, batch number, and composition were listed in Table 1. Two resin cements were used: one self-etch, one self-adhesive resin cement and one type of ceramic were selected: Shofu Disk HC ceramic-based CAD/CAM restorative.

A review of the literature on ceramic bonding showed that 6 to 10 specimens in each group is a widely used sample size. For the power analysis a similar study was used. It showed that a sample size of 4 specimens in each group was enough to obtain 90% power in determining differences between group means [10]. Thus, a sample size of 10 specimens in each group was enough (Power more than 99%).

Teeth preparation

This study was done after the approval of Research Ethics Committee of Future University

in Egypt (FUE.REC (7)3-2023). Forty intact, caries free molar teeth were used. Teeth were extracted within 6 months for periodontal reasons. Periodontal curette was used to remove debris and soft-tissue residue. Teeth were placed in 0.1% thymol solution at room temperature for 1 week, and then it was kept in distilled water at room temperature during the study. The buccal enamel was removed with a diamond saw (Isomet, Buehler, Coventry, IL, USA) at low speed with water coolant to expose the dentin. The dentin surface was grounded by 600 and 800-grit disks to obtain a flat standardized surface. The teeth were embedded in autopolymerizing acrylic resin blocks (Acrostone, Acrostone Dental Factory, Egypt) with the buccal surfaces directed upward.

Preparation of ceramic specimens:

Forty circular specimens of 2.5 mm thickness and 2.5 mm diameter were obtained from CAD/CAM hybrid ceramic (Shofu Disc HC) (98 mm x 14 mm) using a low-speed diamond saw (Isomet 1000; Buehler, Lake Bluff, UK) with coolant. Ceramic specimens were divided into two groups of 20 discs each according to the cement applied either self-etch or self-adhering resin cements.

Cementation of ceramic discs to tooth structure

Each ceramic disc was etched for 20 seconds with 5% hydrofluoric acid Ultradent Porcelain Etch (Ultradent Products, Inc., Köln, Germany). Then, it was rinsed thoroughly with water for 20 seconds. It was dried for 20 seconds with oil/water free compressed air. Alcohol (ethanol) was used to clean the surfaces. HC primer (Shofu, Tokyo, Japan) was then applied and dried until it no longer moved. Then, it was light cured for 10 seconds using LED light-curing device (Elipar S10, 3M ESPE, USA) of intensity (1200mW/cm²). The teeth surfaces were cleaned with water for 10 seconds then they were blotted dry with a sponge. The manufacturer instructions were applied for each cement. Half the specimens in each group were kept in distilled water at 37°C for 24 h before the SBS test.

For the self-etch adhesive based cement: Visalys self-etching single-component primer was applied to the exposed dentin surface of the tooth using brush and was rubbed in for 20 sec. Then, it was blown off with air. The dentin surface was dried with a gentle jet of water/ oil-free air. The Visalys Tooth Primer is nonlight curing. After that, Visalys Restorative Primer was applied with a disposable brush onto the ceramic disc surface and was allowed to work in for 60 sec. Then was blown out and dried with a gentle jet of water/ oil-free air. Visalys Restorative Primer is also non-light curing. Visalys cemcore cement was applied with the self-mixing 1:1 automix syringe on the surfaces of the ceramic disc and on dentin surface. The disc was inserted immediately on the tooth surface. Excess cement was removed by a scaler after initial curing for 2 – 3 sec through brief exposure to light (1200 mW/ cm²). After removal of excess, all surface / cement joint was light cured for 10 seconds.

For the self-adhesive resin cement: Dentin surface was blotted dry with cotton pellet until there is no pooling of water to obtain a moist surface. Without delay, thin, uniform layer of cement was applied using gentle pressure to the ceramic disc surface directly from the mixing tip. The disc was immediately seated on the tooth surface. Light curing was done within the first minute after insertion to facilitate cleanup. The cement was cured for 10 seconds. A scaler was used to remove excess cement. Light curing was done for the exposed disc margins for 20-40 seconds.

Artificial Aging

Thermocycling was done for half the number of specimens in each group. THE-1100 thermocycler (SD Mechatronik, Munich, Germany) was used for specimens thermocycling. Each specimen was subjected to 5000 cycles of 5 °C to 55°C water bath with 30 seconds in distilled water and a transfer time of 5 seconds to simulate 6 months of clinical use.

Evaluation of the Shear Bond Strength between Ceramic and Dentin:

(Instron model 3345, England) with the crosshead speed of a 1-mm/min was used for SBS test until specimen failure. The SBS in MPa was calculated by dividing the force needed for failure (Newton) by the surface area (mm²) using the machine software Bluehill Lite 3 (Instron, England).

Microscopic Evaluation of a Failure Mode:

Stereomicroscope (MA 100 Nikon, Japan with Omnimet image analysis software) with x30 magnification was used for Failure mode observations. The type of failure was classified as: adhesive (between ceramic and cement), adhesive between dentin and cement, cohesive or mixed failure.

Statistical analysis:

The mean and standard deviation values were calculated. Kolmogorov-Smirnov and Shapiro-Wilk tests were used to explore normality of the

data. Data had parametric distribution. independent sample t-test was used for comparison between two groups in non-related samples. The interactions between different variables were tested using Two-way ANOVA. The significance level was set at $P \leq 0.05$. IBM® SPSS® Statistics Version 20 for Windows was used for statistical analysis.

RESULTS

The interaction between the different variables demonstrated by Two-way ANOVA analysis showed that, cement type and thermocycling had a statistically significant effect on SBS to dentin. The interaction between the two variables also showed a statistically significant effect $P \leq 0.05$.

The data in table (2) figure (1) showed the effect of resin cement type on SBS of nanohybrid CAD/CAM ceramic to dentin. There was a statistically significant difference in SBS to dentin between (CU) and (VC) groups with and without thermocycling where ($p < 0.001$). In both groups, the highest mean

TABLE (1) Manufacturer, batch numbers, and composition of the tested materials:

| Material | Manufacturer and batch number | Composition |
|--|--|--|
| HC Primer | Shofu, Kyoto, Japan 71640 | -UDMA, MMA, Acetone, Polymerization initiator and others |
| Shofu Disc HC (hybrid ceramic) | Shofu, Kyoto, Japan 0819919 | -UDMA, TEGDMA, Silica powder, Zirconium silicate, Micro fumed silica, Pigments, and others |
| Visalys tooth primer | Kettenbach GmbH & Co. Eschenburg Germany 37412/0822 | -Water, acidic monomer (10-MDP) and HEMA |
| Visalys® Restorative Primer | -Kettenbach GmbH & Co. Eschenburg Germany 37413/0422 | - Adhesive monomers (10-MDP, silane methacrylate, and Ethanol) |
| Visalys Cemcore (VC) is a dual-curing resin cement | Kettenbach GmbH & Co., Eschenburg Germany 37411/3322 | -Approx. 42 vol.% inorganic fillers in the size range 0.2– 20 μ m including ytterbium fluoride. The polymer base consists of aliphatic dimethacrylates. |
| Calibra Universal (CU) dual-cure Self adhesive resin cement | Dentsply sirona, Konstanz Germany 170821 | Phosphoric acid modified acrylate resin; Barium Boron Fluoro Alumino Silicate Glass Urethane Dimethacrylate; Di- and Tri-Methacrylate resins; Organic Peroxide Initiator; Camphorquinone (CQ);, Accelerators; Butylated Hydroxy Toluene; UV Stabilizer; Titanium Dioxide; Iron Oxide; Hydrophobic Amorphous Silicon Dioxide Particles of inorganic filler range from 16 μ m to 7 μ m, average particle size 3.8 μ m, total filler 48.7% by volume. |

value was found in (VC), while the least mean value was found in (CU) groups.

The data in table (2) figure (2) showed the effect of thermocycling on SBS of nanohybrid CAD/CAM ceramic to dentin. There was a statistically significant difference in SBS of the nanohybrid CAD/CAM ceramic to dentin between (Not-thermocycled)

and (Thermocycled) groups in the different resin cement types where ($p < 0.001$). The highest mean value was found in (Not-thermocycled), while the least mean value was found in (Thermocycled) groups. The results of the failure mode showed that, it was adhesive (at the tooth resin interface) in all the groups.

TABLE (2) The mean, standard deviation (SD) values of SBS of different resin cements in (Thermocycled and not thermocycled) groups:

| Variables | Shear test | | | | p-value |
|------------------|------------|------|---------|------|---------|
| | CU | | VC | | |
| | Mean | SD | Mean | SD | |
| Not thermocycled | 1.88 | 0.12 | 6.71 | 0.53 | <0.001* |
| Thermocycled | 0.60 | 0.07 | 2.79 | 0.52 | <0.001* |
| <i>p-value</i> | <0.001* | | <0.001* | | |

*; significant ($p < 0.05$)

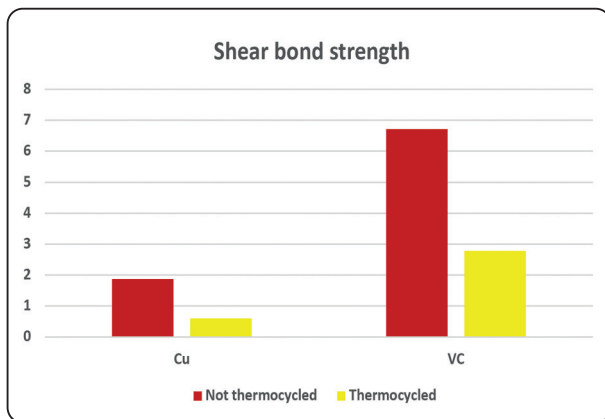


Fig. (1): Bar chart representing the effect of resin cements type on SBS of nanohybrid CAD/CAM ceramic to dentin.

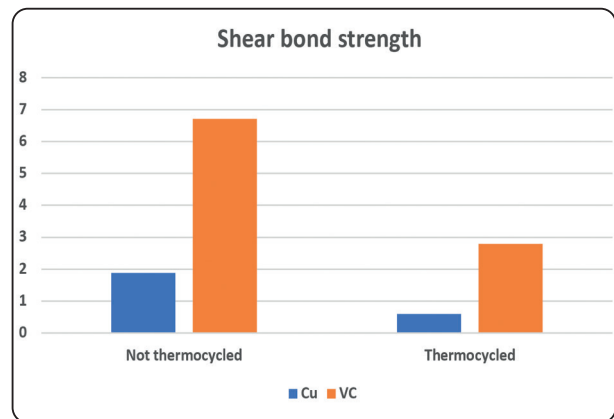


Fig. (2): Bar chart representing the effect of thermocycling on SBS.

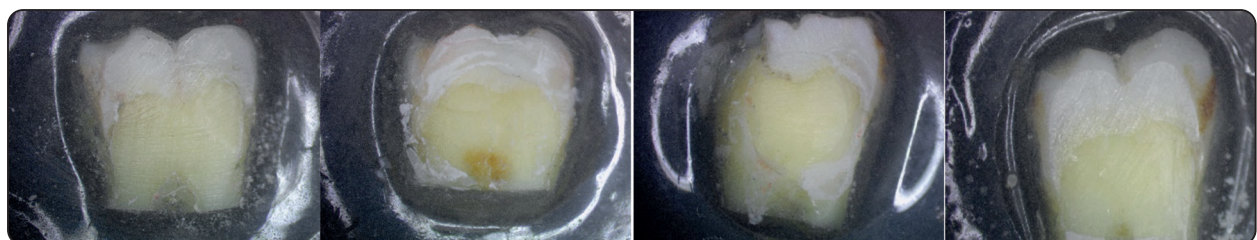


Fig. (3): Stereomicroscope photos representing adhesive mode of failure in different groups.

DISCUSSION

CAD/CAM resin restorations is widely used in dentistry. However, there is conflicting data due to rapid developments in this field, making it hard to decide the perfect surface treatment, and adhesive system for optimum bonding with different types of hybrid CAD/CAM materials. Thus, the current study was aimed to discuss the effect of different resin cements (selfetch adhesive based resin cement and selfadhesive resin cements) on SBS of CAD/CAM nanohybrid ceramic bonded to dentin with and without thermocycling.

The clinical longevity of all ceramic restoration is affected by the strong adhesion of ceramic materials to tooth structure. Many protocols for artificial aging were applied in the literature, such as thermocycling, thermomechanical aging, dynamic load, or water storage [11,12]. In the present study, we used thermocycling. It is a famous accepted methodology for artificial aging of dental materials [13]. Numerous research used the temperature range of 5–55 °C indicated in ISO/TS 11405 Technical Specification for evaluation of adhesion to tooth structure, but they varied in dwell time and number of cycles used [13,14]. A systematic review conducted by analysis of 45 different articles concluded that, artificial aging should be done on 5000 thermal cycles [15]. In the current study, each specimen was subjected to 5 °C to 55°C water bath for 5000 cycles with 30 seconds in distilled water and a dwell time of 5 seconds simulating 6 months of clinical use [16].

In the current study, Shear test was used because it is mostly representative of the clinical situation [17]. The distilled water was used instead of artificial saliva for storage of prepared teeth to simulate the effect of moisture on the resin cements; not the effect of other ions existing in artificial saliva.

The results of this study showed that, different resin cement systems and thermocycling showed significant differences in SBS values of CAD/CAM nanohybrid ceramic bonded to dentin, which led to rejection of the null hypotheses.

The results also showed that, the highest overall values of SBS of the chairside ceramic to dentin in both groups (the thermocycled and not thermocycled group) were for the dual cured self-etch adhesive based cement VC. The lowest overall values of SBS before and after thermocycling were for CU self-adhesive cement. In the research articles, there was contradictory results related to the strength of self-adhesive and adhesive resin-based cements. Some studies showed close bond strength values of both systems and others revealed low bond strength for self-adhesive cements [18, 19,20]. While only a study revealed better sealing for self-adhesive cement in comparison to self-etching cements for ceramic partial crowns [21], another study concluded that, self-etching adhesive cement showed better sealing of the margins than self-adhesive cement, whatever the CAD/CAM material used. [22]

The low SBS of self-adhesive resin cement CU may be related to the fact that self- adhesive cements are not smear layer removing adhesives. This may decrease the resin cement /dentin bond strength. It may be also related to its weight percentage of fillers (wt%), which may affect its viscosity, flow, and infiltration. This is supported by other studies who found that self-adhesive cements act superficially on the tooth structure because it does not dissolve the smear layer [23, 24]. This may be related to the fact that, self-adhesive cements need some pressure during application, to ensure that the relatively high viscosity cement adapted to the surface [25]. Also, self-adhesive cements maintain a low initial pH for a long period which can negatively affect bonding to dentin [26].

The results also showed that, thermocycling decreased the SBS of both cements. This may occur due to hydrolytic degradation and thermal fluctuations. Thermal stresses occur at the interface between the adhesive cement and the ceramic because of variations in thermal expansion and contraction coefficient [27]. Thermocycling also

causes water sorption. The small molecular size of water and the high concentration can penetrate nanometer-size free-volume spaces between polymer chains producing polymer plasticization and leads to decrease in bond strength.^[28]

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The highest SBS values of VC before and after thermocycling may be related to its dual curing mechanism. Obviously, dual curing contributes to increase in bond strength due to higher degree of polymerization in comparison to self-curing or light curing individually. This high degree of polymerization occurs as it contains both initiators present in light-cure and self-cure adhesives^[29]. A proper degree of polymerization of the resin cement translates into better stability at the adhesive interface and contributes to improve durability^[30].

However, in the current study it did not work with Calibra universal self-adhesive cement because of its composition. Calibra universal as a self-adhesive cement contains acidic monomers to achieve surface demineralization to enamel and dentin. These monomers may cause inactivation of the conventional organic initiators, such as benzoyl peroxide/aromatic tertiary amines, negatively affecting both the chemical and light polymerization process^[31,32]. This traditional initiator occurs in Calibra universal cement, possibly contributing to its low bond strength and durability after thermocycling.

In the present study, CU self-adhesive cement failures were adhesive, and were located along the resin/dentin interface. This is in line with Braga et al., who found that, self-adhesive cements commonly show mixed and adhesive failures, and rarely show cohesive failures^[33]. These results are also supported by other studies^[34,35]. These findings agree with the concept that self-adhesive cements interact superficially with the tooth structure.

Regarding VS resin cement despite the higher bond strength values, the mode of failure was also adhesive at resin dentin interface. It is well known that, when there is more than one interface the failure begins at the weaker one.^[36] In the current research, the dentin/resin cement interface was weaker than the resin cement/ceramic interface in all the tested groups. This is because successful bonding of hybrid ceramic is greatly affected by micromechanical retention and chemical bonding. Shofu Nanohybrid CAD/CAM is partially composed of ceramic and partially of resin. Hydrofluoric-acid etching may act on the ceramic part modify the surface micro-structure (partially dissolution of the crystalline phase) creating a micro-porous surface, so improves micro-mechanical bonding with resin cements^[37]. This is in line with another study evaluated the effect of surface treatment on bond strength of polymer infiltrated ceramic network (PICN) and composite CAD/CAM materials after 6 months of artificial aging. They found that, sandblasting and hydrofluoric acid combined with silane showed the best results. These results may suggest that hydrofluoric acid may be a favorable surface treatment with shofu discs HC when using self-etch or self-adhesive resin cements.

However, this study has some limitations, it is difficult to simulate all the oral environment thermal, chemical, and mechanical factors in the *in vitro* studies. These factors are mandatory for imitating the clinical situation and should be evaluated in other future *in vitro* studies, and the results should also be confirmed by longterm *in vivo* studies.

CONCLUSIONS

Within the limitations of this study, it could be concluded that, Self-etch adhesive based resin cement is preferred over self-adhering resin cement in bonding of Shofu Disk HC nanohybrid CAD/CAM ceramic to dentin. Thermocycling showed detrimental effect on SBS of Shofu Disk HC nanohybrid CAD/CAM ceramic to dentin regardless the cement type.

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