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Adhesive Bond Strength of Resin Cements to CAD-CAM Hybrid Ceramic Materials

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ABSTRACT

The present study was conducted to assess the adhesive bond strength of the resin cements to hybrid:computer-aided design (CAD) / computer-aided manufacturing (CAM) ceramics under the standard surface treatment (hydrofluoric etching). Two types of hybrid ceramic: Vita Enamic (VE) and Lava Ultimate (LU) were used to prepare a total of 30 specimen blocks for each ceramic. Each material was divided into three different subgroups depending on the type of cement used [Rely X Unicem (Gp A), Rely X ARC (Gp B) and GIC (Gp C)] (n= 10). Each specimen surface was polished and treated with hydrofluoric acid (10%). Subsequently, silane coupling agent was applied to the specimen in group A and B. Using a putty mould index, cement was build up on each specimen block. After high intensity light cure of group A and B specimen and settling down of GIC in group C, each block was tested for shear bond strength under a load in universal testing machine. Ten samples from each group were assessed for modes of failure. Data was assessed using analysis of variance and Tukey multiple comparisons test. Comparison between the cements in each hybrid ceramic revealed that the highest mean value was for groups B-VE (18.22 ± 1.25) and B-LU (16.56 ± 1.31) and least values were for group C-VE (11.44 ±1.84) and C-LU (10.68±2.17). Further comparison between the two types of hybrid ceramics presented VE to have significantly higher ($p<0.05$) bond strength. The mode failure mostly observed was adhesive followed by the cohesive and admixed. The study displayed a significant influence of different types of cements on SBS of CAD/CAM hybrid ceramics ($p<0.05$). Therefore, to achieve maximum adhesive bonding strength among hybrid ceramic materials, a compatible luting cement and surface treatment is critical.

KEY WORDS: VITA ENAMIC, LAVA ULTIMATE, HYBRID CERAMIC, CAD/CAM CERAMICS AND HYDROFLUORIC ACID.

INTRODUCTION

Modern era of restorative dentistry uses the computer-aided design (CAD) / computer-aided manufacturing (CAM) technology for deliverance of swift production and services (Kassem et al., 2012 Li et al 2014). This chair side service allows faster, improved and efficient quality of products. The system has scanners that capture the structures and displays virtual image of the tooth anatomy for the computer system to design exact similar 3 D restoration to be replaced in the mouth (Elsaka, 2016, Awada and Nathanson, 2015). The computer imaging captures the precise marginal outline and presents with an optimum internal fit for the fixed prosthodontics (Awada and Nathanson, 2015).

Currently, a broad range of materials are available for digital manufacturing process. The decision to use the appropriate CAD/CAM material for the restorative

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material is challenging at times. The materials are divided into two categories, ceramics and composites, including aluminium-oxide, yttrium tetragonal zirconia polycrystals, feldspathic glass ceramics, leucite-reinforced glass ceramics, lithium disilicate glass ceramics, and composite blocks (Elsaka, 2015 Barutcigil et al., 2019).

Composite materials are usually softer, easy to mould, finish and adjust and are less abrasive to opposing teeth; however, show increased wear and tear (Kassem et al., 2012). Nevertheless, ceramic has higher esthetics property, more biocompatibility, resistant to discoloration and wear resistance (Awada and Nathanson, 2015). However, these materials are more susceptible to fractures. Therefore, authors have suggested hybrids for ceramics to stabilise the material.Hybrid ceramics are formulated by mixing two types of materials to enhance the properties of the material and longevity of the restoration. The hybrid ceramic consists of a principal ceramic complex (86 weight %) supported by an acrylic polymer meshwork (14 weight %)(Vita Enamic; VITA Zahnfabrik, Bad Sackingen Germany; Lava Ultimate LAV; 3M ESPE, St. Paul, MN, USA), comprising of a highly cured resin matrix, which is heavily filled with nanoceramic particles (up to 80% by weight) and ceramic nanoclusters (Mörmann et al., 2013, Duarte et al 2016).

These are also known as Polymer-infiltrated ceramic network (PICN), a dual phase ceramic and composite combination (Elsaka, 2016). These materials offer improved biocompatibility and translucency with reduced brittleness and increased flexibility (Kassem et al., 2012, Güngör et al., 2016). Adopting the CAD/CAM material accounts for accuracy in restoration and improves the fracture toughness compared to the ceramics. Ceramic prosthesis have conventionally been cemented with water based cements including, glass-ionomer cement or resin-modified glass-ionomers, particularly in case of zirconia ceramic (Egbert et al., 2015). However due to the failure of water based cements, the use of resin-based cements is recommended for long-term retention and durability (Duarte et al., 2016, Selz et al., 2016, Acar et al., 2016).

Resin cements are common adhesives used to cement indirect all-ceramic restorations. A critical factor in the longevity of indirect restoration is the prevention of micro leakage and positive marginal adaptation, which depends on the adhesive bond strength (Cekic-Nagas et al., 2016). Clinical trials conducted to assess bond strength, has revealed that majority of failures were outcomes of fragmenting, fracture, secondary caries, and debonding of cements (Cekic-Nagas et al., 2016, Flury et al., 2016). Multiple techniques and surface treatments to increase the surface energy creating durable bonds have shown to improve bonding outcomes (Elsaka, 2015, Güngör et al., 2016). Hydrofluoric (HF) acid is considered an effective method for chemical and micromechanical retention of indirect ceramic restorations to cements (Barutcigil et al., 2019).

HF acid dissolves the glassy phase ceramic to create minute interlocking retentive areas (Peumans et al., 2016). Moreover, silane coupling agents are applied to increase the surface wettability through the formation of silane covalent bonds between silica particle in ceramic and methacrylate groups of resin cements (Peumans et al., 2016). At present in current literature, limited data is available in relation to the bond strength of resin cement to hybrid CAD/CAM ceramics. It is hypothesized that no significant difference between the shear bond strengths of the different cements bonded to the hybrid CAD/CAM ceramics will be observed. Therefore, the aim of the present study was to evaluate the adhesive bond strength of the resin cements to the hybrid CAD/CAM ceramics under standard surface treatment.

MATERIAL AND METHODS

The present study was conducted after the approval from the institutional board to demonstrate the adhesive bond strength of resin cements to hybrid CAD-CAM ceramic material. The study uses two types of hybrid CAD/CAM ceramics namely, Lava Ultimate (LU) and Vita Enamic (VE).

Specimen preparation: 30 specimens of each material; group A (VE) and group B (LU) with dimensions 10x10x2mm were prefabricated into clear cut rectangular blocks using a diamond saw (Isomet 1000; Buehler Ltd., Lake Bluff, IL) under a water coolant. Each block was embedded into the acrylic resin dough (Panacryl, Arma Dental, Istanbul, Turkey) to create a flat base for the specimen block followed by carbide polishing. One surface of each block was prepared for a wet ground surface suitable for attachment using a 600 grit silicon carbide (SiC) paper. Subsequently, the specimens were stored in distilled water for 24hrs. Each prepared block surface was etched with 10% of HF acid (Angelus Dental, Londrina, Brazil) for 2 minutes, rinsed in cold distilled water and air dried for 1 minute. Thirty specimens for each hybrid ceramic material (VE and LU) were divided into a total of three groups based upon the three types of cements [Rely X Unicem (Gp A), Rely X ARC (Gp B) and GIC (Gp C)] used (n=10). The materials used in this study are presented in Table 1.

Each group was divided into three Subgroups:Group A-VE: Specimens were coated with a silane coupling agent (SingleBond Universal; 3M ESPE, St.Paul, MN), applied with a microbrush for 60 seconds over the etched surface followed by an air dry for 10 seconds. After the surface preparation, the Rely X unicem cement is build-up using a teflon mold (2 mm diameter, 4 mm thickness). The cement was polymerised with a high-intensity light cure (3M ESPE, St. Paul, USA) for 20 s on each side (total 40 secs) for the RelyX Unicem groups, unit calibrated at 1,900 mW/cm^2.

Group B–VE: Similar procedure was followed as group A-VE; however, the light cure polymerization was for 40 secs (160 secs total) for each side for the Rely X ARC. Group C–VE): Glass-ionomer cement (Vivaglass Cem, Ivoclar Vivadent AG) was homogenously mixed and smeared onto the treated surface of each specimen

Table 1. Materials, composition and manufacturer details.

Material	Composition	Product description
Vita Enamic	Vita Enamic (75 wt% Hybrid feldspar ceramic (resin infiltrated ceramic network) : silicon dioxide 58–63%, aluminum oxide 20–23%, sodium oxide 9–11%, potassium oxide 4–6%, boron trioxide 0.5–2%, zirconia and calcium oxide. Polymer part (25%): UDMA and TEGDMA	Vita Enamic; Vita Zahnfabrik, Bad Sackingen, Germany
Lava Ultimate	Lava Ultimate nanoceramic particles (80%) comprising of silica nanomers (20 nm), zirconia nanomers (4–11 nm), nanocluster particles , progressively cured resin matrix (20%) BisGMA, Bis-EMA, UDMA and TEGDMA and silane coupling agent.	(3M-ESPE, Seefeld, Germany)
RelyX ARC	PASTE A: Silane-treated ceramic, TEGDMA, BisGMA, silane-treated silica, functionalized dimethacrylate polymer, triphenylantimony PASTE B: Silane-treated ceramic, TEGDMA, BisGMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide	3M™ Clicker™ Dispenser, USA.
RelyX Unicem	Base paste: silane-treated glass powder, 2-propenoic acid, 2-methyl-, reaction products with 2-hydroxy-1,3-propanediyl dimethacrylate and phosphorus oxide, TEGDMA, silane-treated silica, sodium persulfate, glass powder, tertbutyl peroxy-3,5,5- trimethylhexanoate, cooper acetate monohydrate 588286 Catalyst paste: Silane-treated glass powder, substituted dimethacrylate, 1-benzyl-5-phenyl-barbic-acid, calcium salt, silane--treated silica, sodium	Aplicap™ / Maxicap™, 3M ESPE, USA.
GIC	Liquid: Polialquenoic acid, tartaric and water. Powder: fluorosilicate glass, Al-Ca-La polymer (5% acrylic acid and malic acid).	3M/ESPE , St. paul, USA.
Universal bonding agent (Silane coupling agent)	10 Methacryloyloxydecyl dihydrogen phosphate, HEMA, silane, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators	SingleBond Universal; 3M ESPE, St.Paul, MN

and allowed to air dried before the application of the load.Group A- LU: A similar procedure to group A-VE was performed for build-up using RelyX Unicem on LU samples.Group B-LU): A similar procedure to group B-VE was performed for build-up using Rely X ARC on LU samples.Group C-LU: A similar procedure to group B-VE was performed for build-up using GIC on LU samples.

After the completion of polymerisation process, the cement was allowed to set before the removal of CAD/CAM resin-ceramic hybrid-composite resin from the mould. Subsequently, the bonded specimen blocks were placed in a water bath for thermocycling. The blocks were thermocycled for 3000 cycles at 5°C and 55°C for a 20 seconds dwell time in a thermocycler (MTE 101; MOD Dental, Esetron Smart Robotechnologies, Ankara, Turkey). Each block was secured with the help of a jig for the shear bond strength testing using a universal testing machine (Lloyd LF Plus; Ametek Inc., Lloyd Instruments, Leicester, England). Each specimen was subjected to a standard force at 0.5 mm/min crosshead speed untill failure. The recorded failure at a maximum load was measured in Newton (N) which was divided by bonding surface area to calculate the shear bond strength in megapascals (MPa). The debonded surface was visualised through a stereomicroscope (DV4; Stemi, G¨ottingen, Germany) to identify the fracture pattern.

Failure mode classification includes three distinctive types namely: type I, adhesive failure, debonding at the interface; type II, mixed failure partially hybrid ceramic, partially resin cement, consisting of both parts and type III, cohesive failure, fracture occurring in the cement. Data were statistically analysed through the statistical program for social science (SPSS). Normality was assessed using Kolmogorov–Smirnov test. Shear bond strength was analysed and tabulated using one-way ANOVA and Tukey multiple comparisons test ($\alpha = 0.05$).

RESULTS AND DISCUSSION

The assessed data was normally distributed. The maximum shear bond strength was observed in group B –VE [18.22 (1.25)], whereas the minimum shear value strength was exhibited in specimens of group C–LU [10.68 (2.17)]. A significant difference was observed among the outcomes of cements (Gp A, Gp B and Gp C) within the hybrid ceramic groups (VE and LU) ($p<0.05$) (Table 2). An overall significant difference was observed among the shear bond strengths between hybrid ceramic materials (VE and LU) ($p<0.05$) (Table 2).

Table 2. Means and SD for shear bond strength among the study groups.

Study groups	VE	LU	ANOVA
Rely U-Gp A	16.28 (1.68)$^{A\ a}$	14.37 (1.47)$^{B\ a}$	
Rely ARC-Gp B	18.22 (1.25)$^{A\ b}$	16.56 (1.31)$^{B\ b}$	<0.01
GIC- Gp C	11.41 (1.84)$^{A\ c}$	10.68 (2.17)$^{A\ c}$	

*Dissimilar superscript capital letter in same row show significant difference
*Dissimilar superscript small letter in same column show significant difference
* Tukey multiple comparisons test

Comparing the two types of resin cements Rely X ARC and Unicem; the shear bond strength value measured was higher in the Rely X ARC in both hybrid ceramic groups. However, GIC showed very low shear bond strength in comparison to resin based cements. Nevertheless, there was a prominent difference between all three types of cement (Rely-X Unicem, Rely-X ARC and GIC) in each CAD/CAM ceramic; VE (16.28 vs 18.22 vs 11.41) and LU (14.37 vs 16.56 vs 10.68) respectively. Moreover, assessment between the CAD/CAM groups showed there was a significant difference between means of SBS for VE and LU for resin cements [Rely-X ARC (VE 18.22 vs LU 16.56) and Rely-X Unicem (VE 16.28 vs LU 14.37)] respectively. However, SBS for VE and LU when bonded to GIC (VE11.41 vs LU .68) was comparable ($p>0.05$).

Table 3. Distribution of failure modes in the tested groups.

Study Groups	Adhesive (%)	Cohesive (%)	Mixed (%)
Gp A-VE	70	0	30
Gp B- VE	50	10	40
Gp C-VE	80	0	20
Gp A-LU	60	10	30
Gp B-LU	20	50	30
Gp C-LU	30	50	20

The modes of failure as observed among the study groups are presented in Table 3. Adhesive failure was most commonly observed among study groups [Gp C–VE (80%), Gp A–VE (70%), and Gp A-LU (60%). However, specimens in Gp C-VE and Gp B- LU both demonstrated 50% of the cohesive failures in cement. Admixed failure mostly ranged from 20–30% except for Gp B-VE, which showed 40% admixed failures. The lowest adhesive failure was noted in Gp B- LU (20%). The present study assessed adhesive bond strength of resin cements to the CAD/CAM hybrid ceramics using standardized technique. Shear bond strength outcomes for different cements on hybrid ceramics were significantly different. Therefore, the hypothesis that different cements will show comparable bond strength outcomes with hybrid ceramic materials was rejected. The study outcomes suggested that the resin cements exhibited comparatively better shear bond strength than the GIC cements. Furthermore, a comparison between two types of hybrid ceramics displayed better adhesive bond strength outcomes for VE than LU. Multiple factors including material properties, adhesive potential, material composition and surface topography are implicated for the observed outcomes.

For the validation of the present study outcomes, thermocycling was performed to age the specimen and mimic oral conditions that are responsible for compromising the adhesive bond strength among tested materials (Campos et al, 2016). The comparison between polymer-infiltrated ceramic materials, Vita Enamic and Lava Ultimate, exhibited a stronger adhesive bond for the former material, particularly with Rely-X ARC (18.22 MPa) cement. This difference in adhesive bonding is explained by the difference in the modulus of elasticity among the restorative materials. Previous studies have specified that the modulus of elasticity (MOE) of LU (12.8 GPa) and VE (30.1 GPa) are closer to the MOE of dentine and resin cement (16 - 20.3 GPa) (Lawson et al., 2016, Belli et al., 2017). The similarity in the MOE allows the homogenous distribution of stress under loads; hence allows long-term retention of indirect restoration withstanding continuous load over a longer. In addition, the resilient polymer matrix base of both hybrid ceramics, exhibits the phenomenal capacity to bond with the resin cement (Belli et al., 2017).

Previous studies have recommended separate surface treatment for each type of hybrid ceramic i.e. HF acid etching for VE and sandblasting for LU (Güngör et al., 2016, Barutcigil et al., 2019). VE contains 86 weight % of feldspar in ceramic filler, which preferably dissolves on application of HF acid compared to LU (80 weight % nanoceramic (Andrade et al., 2018, Sabri et al , 2016). The microstructure gets altered due to the partial dissolution of the polymer and feldspar ceramic glass phase by the acid thus forming micro porosities (El-Damanhoury and Gaintantzopoulou, 2018). By contrast, LU has a high content of 80% silica and zirconia nanoparticles with hard and rough texture which is resistant to surface treatment. This is a possible explanation for the lower bond strength of LU samples in the present study (Andrade et al., 2018).

Earlier studies have assessed the adhesive bond strength by comparing the different surface treatment in the hybrid ceramic materials (Barutcigil et al., 2019, Güngör et al., 2016). Micromechanical retention was suggested as a priority for improvement in the retention of the restorations. Application of hydrofluoric acid as a standard surface treatment for all specimens created a baseline to evaluate the impact of individual cement. The results revealed higher bond strength in the resin cement groups because the acid etching dissolves the glassy particles to create interlocking areas over the surface like a honeycomb (El-Damanhoury and Gaintantzopoulou, 2018).

In addition, the application of the silane coupling agent increases the surface energy for bonding. The outcomes of the present study exhibited better adhesive bond strength for Rely-X ARC than other cements (Rely-X Unicem and GIC). It is known that the silane containing a universal adhesive system is responsible for greater bond strength. Cement selection is the most critical factor in adhesion between the indirect restoration and the tooth structure. Resin cements have demonstrated high bond strength with micromechanical retention grooves aiding in adhesive bond strength (Secilmis et al., 2016). Studies have pointed out that conventional cements such as the zinc phosphate and GIC have a limited capacity for adhesion; however, certain authors recommended zinc phosphate because authors observed that zirconia based cements form a bond with only MDP containing resin cement (Peumans et al., 2016, Duarte et al., 2016).

Thus it limits the maximum bond strength formation. The present study compares resin-based cements (Rely X Unicem and ARC) to conventional cement (GIC). Rely X Unicem is known for its ease in application and low technique sensitivity. The base paste of the Rely X Unicem contains methacrylate monomers comprising of acid phosphoric groups that exhibit self-etching property (Weyhrauch et al., 2016). The property is evident on complete ionisation in a water medium that is present within the paste. The alkaline environment of the catalyst paste allows for neutralisation reaction leading to low pH and low surface interaction leading to the formation of the hybrid layer (Weyhrauch et al., 2016). This hybrid layer allows a desirable bond strength at the interface; nevertheless, the diffusion is limited, which created a lower adhesive bond strength compared to total-etch Rely X ARC with low solubility and high mechanical properties.

Shear bond strength test is the most commonly used bond assessment method in the literature (Flury et al., 2016). Shear bond strength testing in the present study displayed a uniform and homogenous distribution of stress under the load. However, specimen preparation is considered a challenge, as construction of blocks can result in irregularities in small-unbounded area. This could be observed in the form of cohesive failure. The majority of the cohesive failures were observed in Gp C-LU and Gp B–LU, which signifies the material strength. This could also be because of the inaccuracies during the procedure. Despite the fact that aging of the material compromises the adhesive bond strength; Gp B–VE group samples revealed lesser adhesive bond failures, which indicates that the adhesive bond strength was higher than the others. In conventional cementation, GIC does not require pre surface treatment before the application; hence, no interlocking mechanism and limited silane bond formation lowers their adhesive bond strength in comparison to resin cements. (Jevnikar et al, 2012; Sayam et al, 2017)

The present study does posses few limitations that are required to be addressed in future studies. Firstly, there was no variation in the pre surface treatment to distinguish the performance of the cements under various surface roughness with different cement choice. Secondly, the trial performed was relevant to only the two types of CAD/CAM hybrid ceramic. Further trials are necessary to assess the capacity for adhesive bond strength among other contemporary materials. Lastly, there were few of the specimens that displayed cohesive failures, which adds discrepancy; therefore, the adhesive bond may need further evaluation. Therefore, it is recommended to conduct further studies with considerations given to the polymerisation shrinkage and evaluation of surface topography of hybrid ceramics after surface treatments.

CONCLUSION

Within the limitation of the study, it is concluded that the type of CAD-CAM hybrid ceramic material has a significant influence on the quality of adhesive interface it produces with resin, resin modified glass ionomers and glass ionomer cements. Therefore, to achieve maximum adhesive bonding strength among hybrid ceramic materials, a compatible luting cement and surface treatment is critical.

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