Effect of different resin cements on shear bond strength of CAD-CAM crowns fabricated from hybrid materials.

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Declaration

I hereby declare that “An in-vitro study of the effect of different resin cements protocol on the shear bond strength of a CAD/CAM hybrid material” is my own personal work, that it has not been submitted previously in its entirety or in part for any degree or examination at any other University, and that all sources that I have used or quoted here have been properly indicated and acknowledged by a complete list of references.

Mona Zayed Mona

19 November 2020

Dedication

To my husband, Mubarak who supported me in this journey, encouraged me all the way and made this endeavour possible

To my daughter Bibi and my son Rakan, for their understanding, and unconditional love. May God bless them all.

To all my family members and dear colleagues for their great help and support during the accomplishment of this study.

Abstract

Background. CAD/CAM crowns have become popular due to the many advantages associated with this technology. Optimal bonding adhesion is crucial for the durability of these indirect restorations and many factors influence this crucial step. Currently, there is no consensus or evidence-based guidelines on the best adhesion protocol for CAD/CAM crowns fabricated from hybrid materials. This study was aimed at investigating the influence of three types of resin cements on the shear bond strength of a hybrid CAD/CAM material *in vitro*.

Material and methods. One commercially available hybrid CAD/CAM material was subjected to bonding using three different resin cement types: light-cured adhesive cement (n=20), self-adhesive (n=20), and dual-cured adhesive cement (n=20). Shear bond strength and failure mode for each group were compared.

Results. RelyX^{TM} Veneer presented the highest shear bond strength (13.87 ± 1.85 MPa), followed by Rely X^{TM} (10.75 ± 1.43 MPa) and G-CEM Link Force (9.42 ± 1.78 MPa, $p<0.0001$). Adhesive failure was the most frequently observed failure mode, corresponding to 60% in the RelyX Veneer Cement group, 50% in the RelyX U200, and 45% in the G-CEM Link Force. The least common failure mode was the mixed failure, observed in 5% of the RelyX Veneer Cement group, 15% in the RelyX U200 group, and 25% in the G-CEM Force Link group.

Conclusion. Different cement types can influence the adhesion of a hybrid CAD/CAM material. The light-cured adhesive cement presented the highest average values for shear bond strength, followed by self-adhesive, and dual-cure adhesive cements. Adhesive failure

was the most common failure mode in the present study, with the highest frequency in the light-cured adhesive cement group.

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Table of Contents

Table of Contents

List of figures

List of tables

Keywords

CAD/CAM crowns

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Light cure cements

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CHAPTER 1

Literature Review

Introduction

The need for time- and cost-effective aesthetic restorations has driven the advancement of new dental materials and technologies. The concept of computer-aided design and computer-aided manufacturing (CAD/CAM) initiated approximately 25 years ago, offering advantages such as more stable materials, increased productivity, faster treatment turnaround, increased quality, and patient comfort. Further development of this technology has led to the rapid growth of CAD/CAM use in clinical practice in the last decade. CAD/CAM dental restorations can be fabricated from blocks made from ceramic or hybrid materials, which have ceramic and resin composite components (Kömürcüoğlu et al. 2017).

The longevity of indirect dental restorations is highly dependent on the cementation protocol with resin cements. The adhesion interface between the CAD/CAM restoration and the cement is influenced by multiple factors, including restorative material, surface treatment, and chemical characteristics of the cement. Several chemical and mechanical surface treatments for CAD/CAM restorations have been investigated, including the use of alumina, acid-etch, silane, sandblasting, and air abrasion (Spitznagel et al. 2016). Multiple resin cements are commercially available, including conventional, self-adhesive, and adhesive-based options. Some resin cements require light polymerization, while others have a dual-cure. In terms of surface etching, some require self-etch, others require total-etch treatment (Bellan et al. 2017).

Manufacturer's recommendations for cementation are somewhat general and typically include different surface treatments, and use of a coupling agent, such as a silane, before applying the resin cement itself. With so many variation factors involved in the cementation of CAD/CAM restorations made from hybrid materials, there have not been enough scientific studies to provide a consensus or evidence-based guidelines on the optimal adhesion protocol for these restorations (Spitznagel et al. 2016).

Definition of terms

CAD/CAM crowns: crowns that are designed digitally and produced through software for computer-aided design/computer-aided manufacturing.

Hybrid CAD/CAM materials: materials used to fabricate CAD/CAM indirect restorations.

Resin cement: luting material used to perform the cementation of CAD/CAM restorations.

Self-adhesive cement: a cement that does not need preparation of the dental surface through acid and etching procedures.

Dual cure cement: a cement that is cured through light and chemicals.

Light cure cement: a cement that relies on a light source for curing.

Total-etch: a multi-step adhesive protocol that includes acid etching and rinsing of the dental surface.

Self-etch: an adhesive system that has acidic groups in its composition, eliminating the need for acid etching as a separate step.

Shear bond strength: a procedure that evaluates the strength of the adhesion between dental materials and between dental materials and dental tissues.

Review of the literature

CAD/CAM restorations

The development of restorative systems using CAD/CAM has provided a unique opportunity for the fabrication of high-precision indirect restorations in a single appointment. The first system was produced by Werner Mörmann in Switzerland, who created the CEREC® (computer-assisted ceramic reconstructions) system in 1985.

Since then, this technology has evolved, now allowing the fabrication of different types of indirect restorations. With CAD/CAM technology, the prepared tooth is scanned digitally and the restoration is designed virtually using specific computer software. A milling machine is connected to the computer and fabricates the final restoration according to the digital wax-up, built from the digital impression. CAD/CAM restorations are made through the milling of ceramic or hybrid blocks (Mörmann et al. 1987, Kandil 2015).

Some of the advantages of CAD/CAM restorations include adequate aesthetic results, high mechanical durability and predictability, increased efficiency in the laboratory process, and faster fabrication of the restoration. The main limitations are the higher costs and the challenges related to data acquisition through intra-oral scanning. (Miyasaki et al. 2009)

CAD/CAM materials

Highly reproducible manufacturing protocols result in the production of reliable CAD/CAM blocks, which are void-free. Originally, the first CAD/CAM restorations could only be fabricated from glass-ceramic blocks. Nowadays, there are a variety of CAD/CAM materials, including high-strength and silica-based ceramics, resin composite, and hybrid materials. (Spitznagel et al. 2016) CAD/CAM hybrid materials are somewhat new in restorative dentistry, and they can be divided into two main categories according to their composition: those composed mainly by resin matrix (commercially available as Lava Ultimate, Shofu, and Cerasmart) and those composed mainly by ceramics (known as hybrid ceramics, commercially known as Vita Enamic). (Spitznagel et al. 2016).

Table 1. Overview of hybrid materials for CAD/CAM restorations (Source: Spitznagel et al. 2016)

CAD/CAM MATERIAL	MANUFACTURER	CLASSIFICATION	COMPOSITION	SURFACE TREATMENT	ADHESIVE SYSTEM
VITA ENAMIC	VITA Zahnfabrik	Hybrid ceramic	Aluminum oxide enriched, feldspar matrix (86 wt%, 75 vol%) permeated by a polymer (UDMA and TEGDMA, 14 wt%, 25 vol%)	60-second etching with 5% HF	Silane + composite cement
LAVA ULTIMATE	3M ESPE	Resin nanoceramic	Bis-GMA, UDMA, BisEMA, TEGDMA, silica zirconia, silica/zirconia cluster	Air-particle abrasion w ith 50 pm A l20 3(2 bar)	Ceramic primer (silane) + composite cement
SHOFU Block/ Disk HC	Shofu	Ceramic material	UDMA, TEGDMA Silica powder, zirconium silicate, silica, pigments, others	Air-particle abrasion (50 pm A l20 3 at 0.2 - 0.3 bar for 10s)	Ceramic primer + composite cement
CERASMART	GC America	Flexible nanoceramic	Silica, barium glass, Bis-MEPP, UDMA, DMA	Air-particle abrasion (50 pm A l20 3 at 1.5 bar)	Ceramic primer (silane) + composite cement

CAD/CAM blocks vary regarding the composition of the organic matrix, as well as the size, amount, and composition of fillers. According to composition, hybrid ceramics can also be divided into dispersed fillers (DF) and polymer-infiltrated ceramic networks (PICN). (Eldafrawy et al. 2019)

Despite the fact that hybrid ceramics present high wear resistance than resin nanoceramics, their resistance to elastic deformation is higher when compared to dental structures. Cerasmart blocks (GC Europe Lava Ultimate; 3M ESPE), which are a type of nanoceramics, have been suggested to be adequate substitutes to dense ceramics. (Ruse et al. 2014)

Nanoceramic CAD/CAM blocks

The composition of nano ceramic blocks includes a polymeric matrix and a reinforcement in the form of ceramic fillers or nanohybrid fillers. The fabrication process of these blocks is characterized by high pressure and temperature, leading to improved mechanical properties, such as increased conversion rates, volume fraction filler, and modulus of resilience. (Bottino et al 2015) Resin nanoceramic blocks have also been suggested to cause less wear to opposite natural teeth, with higher resistance to chipping and fracture due to a Young modulus that is comparable to dentin. Furthermore, these CAD/CAM materials can be easily polished and repaired. (Tsitrou et al. 2007)

Goujat et al. (2018) compared the internal fit and mechanical properties of Cerasmart (GCDental Products), Lava Ultimate (3M ESPE), Vita Enamic, and IPS (e.max CAD) *in vitro*. The highest flexural strength and best internal fit were observed for Cerasmart and IPS.

In a recent *in vitro* study, CAD/CAM blocks presenting different compositions were analysed for their mechanical properties. When compared to PICN ceramic, resin composite CAD/CAM blocks presented elastic moduli and hardness that were positively correlated to the microstructure and the percentage of ceramic filler. In addition, hardness and elastic moduli of composite CAD/CAM materials were comparable to dental tissues. (Alamoush et al. 2018)

Bonding of CAD/CAM materials

Indirect restorations rely on the robust bond between the restoration and the cement, and between the latter and the dental tissues. The strength and the quality and of the bond ultimately affects the clinical success and longevity of the restoration. Adequate bonding depends on several factors, including the selection of an appropriate cement and suitable surface preparation. (Kurtulmus-Yilmaz et al. 2019)

Adhesive systems

When using adhesive-based cements, adhesive protocols are required in order to strengthen the bonding between the dental surface and the restoration. (Van Meerbeek et al. 2003) There are two main adhesive systems in restorative dentistry, self-etch, and total-etch protocols.

Total etch systems

Also known as etch-and-rinse protocols, total-etch systems involve two separate phases: etching and rinsing. The etching is typically performed through the application of phosphoric acid (30-40%) on dentin, followed by rinsing. In some systems, priming is required after etching (three-step procedure), while in one solution includes both adhesive and primer (two-step procedure). (De Munck et al. 2005)

The effects of total etching systems lead include the elimination of the smear layer, collagen fibres exposure, and demineralization of hydroxyapatite crystals (up to a few micrometres in depth), leading to the creation of micro porosities. Because the adhesive resin has a high viscosity, after application, it penetrates the porosities, and, once polymerised, it forms micro and macro tags that increase retention. For self-etch protocols, the main limitation lies in the sensitivity of the technique, which has multiple steps that increase treatment time. (Van Meerbeek et al. 2003, 2010)

Self-etch systems

In self-etch systems, the adhesive has phosphate acid or carboxylic groups in its composition, reason why it eliminates a few steps as it can etch and prime the dentin surface. These systems cause the dissolution of the smear layer, however, the resulting calcium phosphate is not removed due to the non-rinsing protocol. Reduced exposure of collagen fibres is observed in these systems, which can decrease nano leakage. (De Munck et al. 2005)

According to their acidity and ability to etch, self-etch systems can be categorized as strong, intermediate strong, and mild. (Van Meerbeek et al. 2003) These systems can have one step (without primer) or two steps, which involves the application of a primer before the adhesive. Studies suggest that bond durability and efficiency are increased in two-step self-etch systems. (Tay & Pashley 2003, Van Landuyt et al. 2009)

Resin cements

Given their beneficial mechanical properties, adequate aesthetics, low solubility, and strong bonding, resin cement are commonly used for cementation of non-metallic CAD/CAM restorations. They can bond to dental tissues and restorative surfaces. (Hitz et al. 2012, Ferracane et al. 2011, Weiser & Behr 2015)

Resin cements present low solubility, and optimal mechanical properties, as compared to other types of cement, reason why it has been extensively used for indirect dental restorations (Van Meerbeek et al. 2003). They are typically divided into two categories: one requires the use of an adhesive (adhesive-based cement), and the other does not require adhesive (self-adhesive cement).

Several factors are typically taken into consideration during the selection of a resin cement for indirect restorations, including personal preference, technique, delivery system, shades available, and cost. Another factor to be considered is polymerization, which can be controlled through the application of visible light in light-cured and dual systems. With these systems, working time can be extended to allow for the removal of cement excess, which is not the case for self-cured cements. (Reiss 2006) When comparing light-cured to dual cements, the latter can be advantageous when dealing with thick restorations that could hinder light polymerization. (Garcia et al. 2007)

Self-adhesive dual-cured resin cements reduce many clinical steps, potentially decreasing moisture and post-operative sensitivity. (Broyles et al. 2013, Burgess et al. 2010) When using this type of cement, micro-mechanical surface retention is achieved through the contact between acidic monomers and dentin, causing demineralization and penetration of the luting agent into the dental substrate. Furthermore, chemical retention can further increase bonding strength due to the reaction between hydroxyapatite and phosphoric acid components. (Pisani-Proenca et al. 2011) Nevertheless, limited etching capacity, low bond strength, inadequate adhesion to dentin, high cement viscosity, and low penetration have been described as limitations of resin cements that do not require the use of adhesives. (Pavan et al 2010, Santos et al. 2011)

Adhesive based cements

Adhesive-based cements can also be described according to the adhesive system as total etching and self-etching (Hitz et al. 2012, Hill 2007). Adhesive-based resin cements can be classified as dually cured, light-cured, or self-cured in terms of polymerisation requirements. (Manso et al. 2011) For dual-cure adhesive cements, it is recommended that light is only applied after giving the mix enough time for the self-curing process to take place. (Pegoraro et al. 2007)

Shear bond strength analysis of different curing modes for resin cements requiring revealed no significant difference between light- and self-curing systems when used in CAD/CAM composite blocks. (Kim et al. 2016) However, other studies suggest that self-curing is less effective than dual-cure cements in terms of rate of polymerization, bond strength, cement hardness, conversion, and solubility, which can compromise mechanical properties, and potentially decrease the long-term success of indirect restorations. (Arrais et al. 2008, Luhrs et al. 2014, Killinc et al. 2011)

In general, adhesive resin cements present the advantages of predictability and high bond strength. They can be used in a variety of clinical scenarios, including full and partial restorations. (Blatz et al. 2003) The limitations include sensitive technique, difficult removal of excess cement, and potential for marginal discoloration, which can become a problem in aesthetic areas. (Spitznagel et al. 2016)

Self-adhesive cements

Self-adhesive cements were developed more than 20 years ago as an easier alternative to the multi-step process of conventional adhesive-based cements, as they eliminate the need for the use of an adhesive and etching. Hence, the dental tissues do not need to be prepared before being subjected to the application of the cement. The dimensional stability, simplicity of application, optimal mechanical properties, and the maintenance of the smear layer are the major advantages for this type of cement (Makkar & Malhotra 2013). Self-adhesive cements seem to be able to tolerate moisture, and some have the ability to release fluoride. (Weiser & Behr 2014)

Currently, all commercially available resin cements that require a self-adhesive protocol are radiopaque and dually cured, which allows for use with composite, ceramic and metallic indirect restorations. Etching of the tooth surfaces is possible due to the presence of methacrylate monomers with functional acidic groups (carboxylic or phosphoric groups) in the composition of the cement. (Makkar & Malhotra 2013, Radovic et al. 2008) Initially, during cementation, self-adhesive cements are hydrophilic, however, they become gradually more hydrophobic as curing progress, resulting in micromechanical interlocking and chemical bonding. (Ferracane et al. 2011)

Comparison between self-adhesive and adhesive based cements

In the literature, there have been conflicting results regarding the strength of self-adhesive and adhesive resin-based cements, with some studies advocating comparability between the two systems and others suggesting inferior adhesion strength for self-adhesive cements (De Munck et al. 2004, Abo-Hamar et al. 2005, Goracci et al. 2006, Al-Assaf et al. 2007, Hikita et al. 2007).

Hikita et al. (2007) have suggested that when application protocols are strictly followed, self-adhesive and adhesive-based cements present similar results when the adhesion dental tissues are investigated. Weaker adhesion to enamel has been associated with the use of self-adhesive cements. (Radovic et al. 2008)

When fracture resistance was evaluated, feldspathic CAD/CAM crowns cemented with a self-adhesive system presented better results than cements that require etching and rinsing. (Mornann et al 2009) However, a similar study found comparable results in fracture resistance when comparing these two cement systems. (Burke et al. 2006)

With regards to nano leakage, comparable results have been reported for ceramic CAD/CAM materials using total-etch or self-adhesive cement. (El Badrawy et al. 2011)

Marginal sealing has also been evaluated and studies report contradictory results. While one study showed better sealing for self-adhesive cement when compared to self-etching cements for ceramic partial crowns (Schenke et al. 2008), another research showed that self-etching adhesive cement presented improved marginal sealing than self-adhesive cement, irrespective of the CAD/CAM material used. (Ghazy et al 2010)

One study compared several combinations of luting agents and surface treatments used in four different CAD/CAM resin materials (Kömürcüoğlu et al. 2017). The results showed that sandblasted surfaces and surfaces etched with hydrofluoric acid used with a universal adhesive resulted in the highest bond strength.

In a study from Bellan et al. (2017), a few resin cements were compared for different brands of CAD/CAM milled resin crowns cemented to dentin. Comparable micro tensile bond strength was reported for conventional and self-adhesive resin cements. Ishii et al (2017) reported that, for *in vitro* cementation of CAD/CAM onlays with an adhesive cement, immediate dentin sealing using an all-in-one adhesive protocol and flowable composite led to greater bond strength.

Surface treatment

The bonding of artificial restorative materials to vital dental tissues remains a challenge in dentistry. Several types of surface treatment have been developed with the objective of increasing micro-roughness, micro-mechanical retention, and bonding to the resin cement (El-Damanhoury & Gaintantzopoulou 2018). Micromechanical retention and chemical bonding are likely the two most crucial surface treatment parameters for the successful bonding of hybrid materials. The influence of treatment with hydrofluoric etching, silica coating, particle abrasion, and laser on the adhesion strength of resin cements to novel CAD/CAM blocks has been evaluated. When considering surface treatment, the chemical composition of the CAD/CAM material must be considered. Hydrofluoric acid has been generally accepted as the

the gold standard for glass-ceramics. However, for hybrid CAD/CAM materials, there is less clarity regarding surface treatment (Kurtulmus-Yilmaz et al. 2019)

The influence of three different surface treatments (silane application, alumina abrasion, and hydrofluoric acid) on the retention of CAD/CAM restorations cemented with MaxCem Elite and the adhesive Optibond XTR was evaluated by Nejat et al. (2018). All surface treatments improved bonding, the highest results being observed for the alumina treated groups with or without silane (Nejat et. al 2018).

Another recent study investigated the impact of sandblasting with particles of different sizes on the adhesion strength between dual-cured cement and resin CAD/CAM blocks (Cerasmart, Vita, and Lava). One of the brands was not influenced by particle size (Vita), while the other two presented increased bonds for sand particles with higher size (Tekçe et al. 2018).

Tian et al. (2014) suggested that surface treatment with silane should be recommended for CAD-CAM composites. When applied to the restoration, silane molecules initially form dimers, which react to form siloxane oligomers. It has been suggested that the mechanism through which silane improves chemical bonding includes increased wettability of ceramic surfaces. (Tian et al. 2014) A recent study analyzed the effect of the brand, air abrasion, and silane or resin primer as the surface treatment for three different CAD/CAM resins. (Reymus et al. 2018) Groups with silane pre-treatment presented the highest failure rates.

Lise et al. (2017) evaluated the impact of surface protocols on the adhesion strength of PICN and composite CAD/CAM materials after 6 months of artificial aging. The absence of treatment led to the worse results and lowest strength, with the best results observed for sandblasting or hydrofluoric acid combined with silane. After six months, lack of treatment and lack of silane resulted in the highest failure rates.

Previous studies indicate that airborne particle abrasion can improve micromechanical interlocking to a higher degree when compared to etching before application of adhesive cements in DF CAD/CAM composites (Spitznagel et al. 2014, Eldafrawy et al. 2018, Reymus et al. 2018, Eldafrawy et al. 2019). Hence, surface preparation through airborne particle abrasion seems to improve the bonding properties of DF CAD/CAM composite materials.

In a review from 2019, which included results from 32 articles, the authors concluded that it is necessary to create micro retention in the surfaces, with sandblasting and hydrofluoric acid etching showing the best results. After this first step, silanization of the CAD/CAM material should follow for improved chemical adhesion before using the resin cement. (Mine et al. 2019)

Clinical studies on the longevity of CAM/CAM restorations

When compared to conventional indirect restorations, the number of well-controlled clinical studies on the longevity of CAD/CAM restorations is not as vast. A systematic review from Wittneben et al. (2009) estimated the 5-year total survival of single tooth CAD/CAM restoration made from glass ceramics to be 91.6%. The total number of restorations included in the review was 1.957, from 16 studies, with a mean follow-up of 7.9 years. The authors did not find a relation between the type of luting cement and survival rates.

Few clinical studies have been published on other CAD/CAM materials. A recent study from Oz et al. (2020) included 38 CAD/CAM nano-ceramic crowns in 26 patients (including vital and non-vital teeth), with a mean follow-up time of 45 months. All crowns were sandblasted and received a layer of silane before cementation with an acid-etch, dual-cure adhesive cement. The observed survival rate was 86.8%. The reason for failure in vital teeth was debonding, and in non-vital teeth, it was tooth or restoration fracture. (Oz et al. 2020)

Similar results were presented by Zimmerman et al. (2018), who reported a 7.1% debonding rate for nanoceramic crowns cemented with dual-cure resin cement, and an overall survival rate of 85.7% after 2 years. Because three out of five failures were due to debonding, the authors highlighted the importance of the bonding protocol to the bonding strength. (Zimmerman et al. 2018)

Conclusion - literature review

CAD/CAM resin restorations have been broadly used in dental practices worldwide, however, science has not been able to follow the quick developments in this field, and currently, there is conflicting data, making it difficult to determine the ideal surface treatment and adhesive system for optimal bond strength when using composite CAD/CAM materials. The many variation factors for cementation and surface preparation, including different methodologies, different commercial products, different protocols, and materials, suggest that further research is necessary to further explore the question of which is the best adhesion procedure to cement CAD/CAM crowns, as measured through shear bond strength.

CHAPTER 2

Statement of the problem

Hybrid CAD/CAM materials offer efficient milling, however, debonding of these restorations can become a problem in clinical practice, affecting treatment longevity, especially for full-coverage crowns. There are a few hybrid materials for CAD/CAM crowns and a variety of resin cements available. Currently, there is no scientific evidence to provide a consensus on the most efficient adhesion protocol for these indirect restorations.

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CHAPTER 3

Aims and objectives

Aims

To assess the impact of one self-adhesive resin cement and two different adhesive resin cements (one light-cured and the other dual-cured) in the shear bond strength of CAD/CAM blocks of hybrid nature *in vitro*.

Objectives

- To investigate and compare self-adhesive, adhesive dual, and light-cured resin cements for a hybrid CAD/CAM material, measured as shear bond strength.
- To investigate and compare the frequency of different failure modes of the above-mentioned cements in relation to one hybrid CAD/CAM material.

CHAPTER 4

Methodology

Design and ethical approval

This study was performed *in vitro* and ethical approval was attained from UWC Health Research Committee and Senate. All aspects of the protocol were designed in accordance with the UWC Research Ethics Policy.

CAD/CAM Material

One hybrid CAD/CAM material was used in this study, which presents the best characteristics of resin composites and high strength ceramics (Cerasmart (GC America, Figure 1). Cerasmart constitutes a flexible CAD/CAM nano-ceramic block with a high density (71%) of barium glass and silica particles surrounded by a highly-cured resin matrix. The polymers found in Cerasmart include Bis-MEPP, UDMA, and DMA (Awada & Nathanson 2015).

2018).

Figure 1. CAD/CAM Cerasmart blocks (GC America).

Specimen preparation

Using a cutting saw, the CAD-CAM hybrid blocks were sliced using a low-speed into rectangular plates (5mm radius × 5mm height, n=20 per group). This process was performed underwater cooling (Isomet 1000, Buehler Ltd, Figure 2), with a total sample size of 60. The prepared specimens were inserted into a customized mold made of silicone (15 mm in diameter × 10 mm thick, Figure 3) and filled with auto polymerizing acrylic resin (Alp et al. 2018).

Figure 2. Low-speed saw used for cutting blocks (Buehler Isomet, 11-1180-160).

Figure 3. Sample blocks (Cerasmart)

For surface standardization, in each specimen, one side was polished underwater using an abrasive paper (silicon carbide 600 grit). The surface treatment was the same for all specimens and followed the manufacturer's instructions: air abrasion for 15 seconds with Al_2O_3 particles (50-µm), at a distance of 10 mm. The surfaces were subjected to ultrasound cleaning and immersed in distilled water for 15 min, followed by air drying. (Alp et al. 2018)

Table 2. Different dental materials used in the study.

MATERIAL	NAME	TYPE	MANUFACTURER	COMPOSITION
Hybrid material	**Cerasmart**	CAD/CAM hybrid	GC America	Silica, barium glass, Bis-MEPP, UDMA, DMA
Resin composite cement	**RelyX™ Veneer Cement**	Adhesive resin cement light cured	3M ESPE	(BisGMA and TEGDMA, Zirconia/silica and fumed silica polymer
Resin composite cement	**Rely X™ U200**	Self-adhesive resin cement	3M ESPE	Base: Methacrylate monomers composed by phosphoric acid groups, Silanated fillers Initiators, Stabilizers, Rheological additives Catalyst paste: Methacrylate monomers basic fillers, Initiators, Stabilizers Pigments Rheological additives
Resin composite cement	**G-CEM Link Force**	Adhesive resin cement dual-cured	GC America	Etching: 37% phosphoric acid, silicon dioxide, colorant G-Multi Primer: 10 MDP, 10 MDTP, 3-methacryloxypropyltrimethoxysilane, ethanol G-Premio Bond: methacrylate acid ester, 10 MDP, 4-MET, distilled water, 10 MDTP, acetone, photoinitiators, silica G-Premio Bond: initiator, ethanol, water, Base: Bis-GMA, UDMA, barium, dimethacrylate, silica and glass filler, initiator Catalyst: UDMA, Bis-MEPP, dimethacrylate, silica and barium filler, initiator, pigment, Filler: 62 volume percentage
Etching agent	**Ultradent Porcelain Etch**	Hydrofluoric acid	Ultradent Products, Inc., Köln, Germany	Hydrofluoric acid 9% buffered
Ceramic primer	**G-Multi primer**	Silane + MDP +MDTP	GC America	Ethyl alcohol, dimethacrylate component, phosphoric acid ester monomer

Ceramic primer	**RelyX Ceramic Primer**	Silane	3M ESPE, Seefeld, Germany	Ethyl alcohol, Water, Methacryloxypropyltrimethoxysilane.

According to the type of resin cement used, the prepared CAD/CAM specimens were divided into three groups:

- Self-adhesive resin cement (Rely X^{TM} U200, n=20, Figure 4)

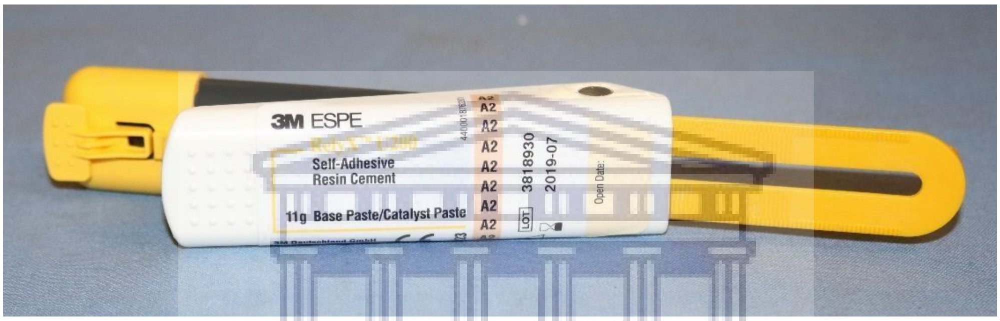

Figure 4. Rely X^{TM} U200

- Adhesive cement light-cured (Rely X^{TM} Veneer Cement, n=20, Figure 5)

Figure 5. Rely X^{TM} Veneer Cement

- Adhesive cement dual-cured (G-CEM Link Force, n=20, Figure 6)

Figure 6. G-CEM Link Force.

Table 3. Different cements and respective protocols used in the study.

CLASSIFICATION	MATERIALS (MANUFACTURERS)	INSTRUCTIONS FOR USE
Adhesive-based resin cement, light cure	Rely X™ Veneer Cement (3M™)	Application of silane (3M ESPE), slightly air-thinned (2-5 seconds), followed by an adhesive resin (Scotchbond Universal Adhesive; 3M ESPE), slightly air-thinned, apply of Rely X veneer cement to the ceramic. Application of cement and light-curing from all directions (20 sec. per surface)
Self-adhesive cement, dually cured	Rely X^{TM} U200 (3M™)	Base and catalyst paste homogeneously mixed during 20 seconds using a spatula, Application of cement.
Adhesive cement, dually cured	G-CEM Link Force (GC America)	Apply G-Multi Primer GC to the surfaces (20 sec), slight air-drying (5 sec), followed by additional light-polymerization (10 s). Apply cement and light polymerize for 20 s at 4 proximal sides (total of 100 s)

Using a customized silicone mould, cylinders made from composite resin were prepared under standardized measurements (4 mm radius × 3 mm height, Figure 7). A hand instrument was used to condense the composite resin, added in incremental layers of 3-mm before being subjected to light polymerization. The bonding area corresponded to the resin cylinder diameter in the center of the specimens. Three types of cements were used to bond the specimens: self-adhesive, adhesive light-cured and dual-cured.

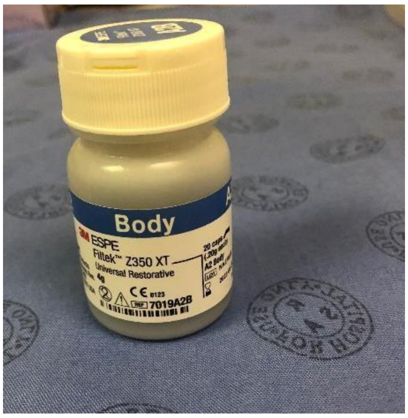

Figure 7. Filtek Supreme XTE (3M ESPE, 2010)

In the light-cured group (Rely X^{TM} Veneer Cement), silane application for 60 seconds and air-drying was followed by the universal adhesive application (Scotchbond; 3M ESPE, Figure 8), slightly air-thinned, apply of Rely X veneer cement to the ceramic.

In the dual-cure group (G-CEM LinkForce GC), before application of the cement, the bonding agent (G-Multi Primer GC) was applied (20 seconds), air-dried slightly for 5 seconds, and light polymerized for 20 seconds.

For Rely X^{TM} U200, comparable volumes of the catalyzer and the base paste were mixed for a total of 10 seconds before application of the cement. Luting was performed by the weight of 100 g using a custom-made alignment apparatus at room temperature.

A brush was used to remove excess cement before the specimens were light polymerized for 20 seconds (each proximal side, total of 100 seconds) (Alp et al. 2018) The light-curing was induced using an LED light unit according to the user instructions, with an irradiance of 1200 mW/cm2 (Elipar S10, 3M Espe, St. Paul, MN, Figure 8). Finally, all specimens were immersed in distilled water for a total of 24 hours at room temperature (Figure 9). (Alp et al. 2018).

Figure 8. LED light unit for curing (Elipar S10, 3M Espe, St. Paul, MN)

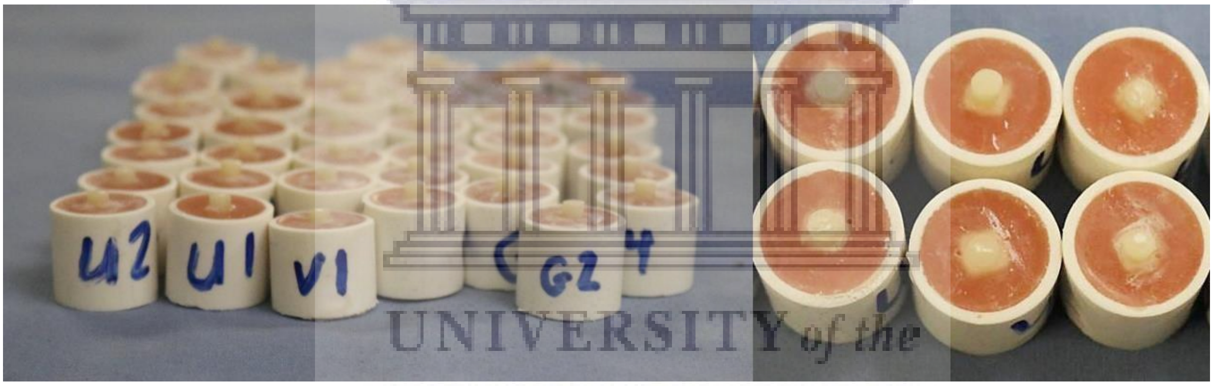

Figure 9. Specimens ready for testing.

Shear bond strength and failure modes

All specimens were subjected to a static shear bond strength test using a universal testing machine (Tinius Olsen H10KT, Horsham, USA). Each specimen was individually placed and secured in a jig, and exposed to a shear load with a blunt knife-edged shearing rod at 1.0 mm/min cross-head until breakdown (Figure 10). Loading was applied perpendicular to the adhesive interface (Figure 11). The angle for loading for the specimens was at 90° to the plate (Figure 12). The values were calculated in Mega Pascals (MPa) according to the formula: maximum load failure (N)/bonding area (mm^2).

Figure 10. Universal machine used for the tests.

The specimens were evaluated under a light microscope under ×10 magnification for failure modes (Wild Heerbrugg M5, Switzerland, Figure 13). Failure modes were categorized as cohesive (when it occurred within composite or resin cement), adhesive (when it occurred between composite and resin cement), or mixed (when there was a combination of adhesive and cohesive modes) (Alp et al. 2018).

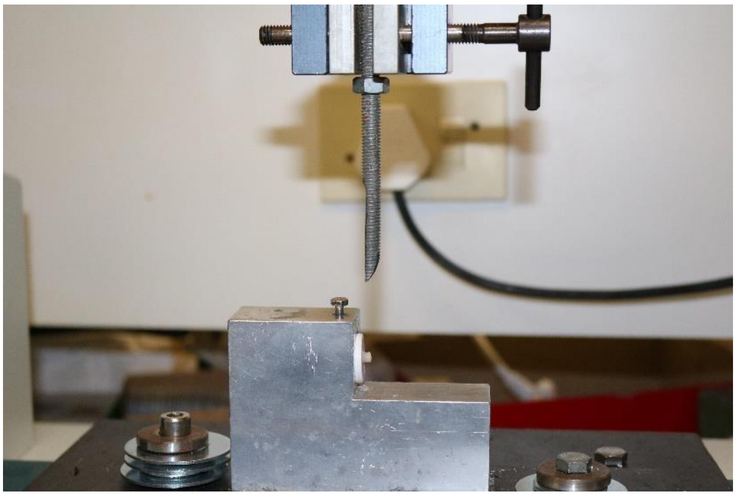

Figure 11. Specimen supported on a Jig.

Figure 12. Loading configuration of the shear bond test.

Figure 13. Light microscope: Wild Heerbrugg M5 (Switzerland)

Data capture and analysis

During the laboratory experiments, all samples were coded to facilitate data capture and to allow blind analysis. All data were captured in spreadsheets (Excel© (Microsoft Corporation, Redmond, WA, USA) and double-checked for eventual mistakes.

Data was described through mean and standard deviation when normally distributed. A one-way ANOVA with pairwise comparisons (and Bonferroni corrections for multiple testing) was used to evaluate differences between the multiple materials. All data were analyzed using Stata (StataCorp LLC Stata Statistical Software 15, 2017, College Station, TX:). Statistical significance was considered for P values below 0.05.

CHAPTER 5

Results

Shear Bond Strength Analysis

The values for the shear bond strength analysis for each group were evaluated (n=20 samples for each group). Table 4 presents average shear bond strength and standard deviation according to the resin cement group.

Average shear bond strength was statistically different for the three study groups, with RelyX^{TM} Veneer presenting the highest value (13.87 ± 1.85 MPa), followed by Rely X^{TM} U200 (10.75 ± 1.43 MPa) and G-CEM Link Force (9.42 ± 1.78 MPa, $p<0.0001$ - Table 4).

Table 4. Shear bond strength (MPa) average values in relation to different resin cements.

MATERIAL	AVERAGE	STANDARD DEVIATION	P-VALUE ANOVA
RelyX™ Veneer Cement	13.87	1.85	**p<0.0001**
Rely X^{TM} U200	10.75	1.43	
G-CEM Link Force	9.42	1.78	

Pairwise comparisons were performed (Bonferroni correction) with statistically significant differences between the RelyX™ Veneer Cement and Rely X^{TM} U200 ($p<0.001$), between RelyX™ Veneer Cement and G-CEM Link Force ($p=0.016$), and between G-CEM Link Force and Rely X^{TM} U200 ($p<0.001$, Table 5 and Figure 14).

Table 5. Bonferroni test results for comparison between different study groups.

GROUP COMPARISON BONFERRONI TEST	MEAN DIFFERENCE	P-VALUE	95% CONFIDENCE INTERVAL	
RelyX™ Veneer Cement versus Rely XTM U200	3.11	**<0.0001**	2.04	4.19
RelyX™ Veneer Cement versus G-CEM Link Force	-1.33	**0.016**	-2.41	-0.26
G-CEM Link Force versus Rely XTM U200	-4.45	**<0.0001**	-5.53	-3.37

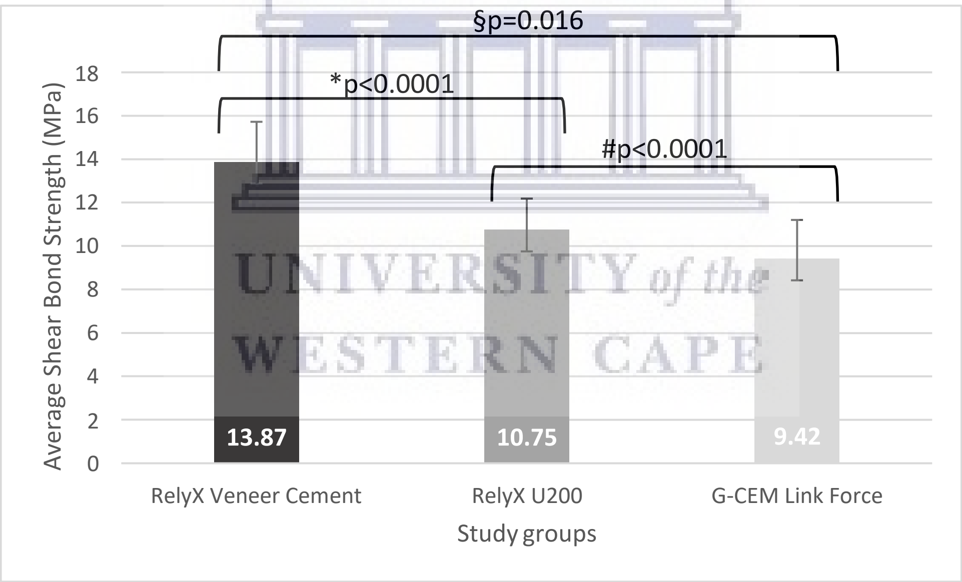

Figure 14. Average shear bond strength (MPa) according to the study group. P-values presented for Bonferroni test between groups: §p=0.016 for comparison between RelyX™ Veneer Cement versus G-CEM Link Force; *p<0.0001 for comparison between RelyX™ Veneer Cement versus Rely XTM U200; and #p<0.0001 for comparison between RelyX™ Veneer Cement versus Rely XTM U200. Vertical bars represent standard deviation.

Failure mode

For all groups, the most dominant failure type was adhesive failure between the ceramic and the cement, corresponding to 60% (n=12) of the failure in the RelyX Veneer Cement group, 50% (n=10) in the RelyX U200, and 45% (n=9) in the G-CEM Link Force.

The second most frequent failure mode was a cohesive failure (failure within the ceramic), representing 35% of the failures in the RelyX Veneer Cement (n=7) and RelyX U200 (n=7), and 30% in the G-CEM Link Force group (n=6).

The least common failure mode was the mixed failure, observed in 5% of the RelyX Veneer Cement group (n=1), 15% in the RelyX U200 group, and 25% (n=3) in the G-CEM Force Link group (n=5, Figures 15 and 16).

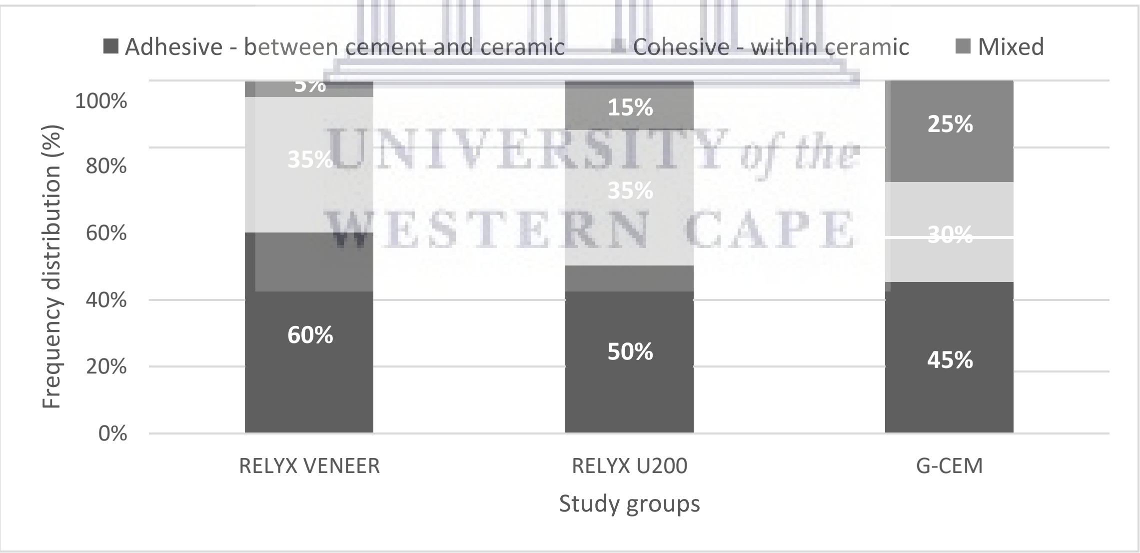

Figure 15. Failure mode (%) according to cement group.

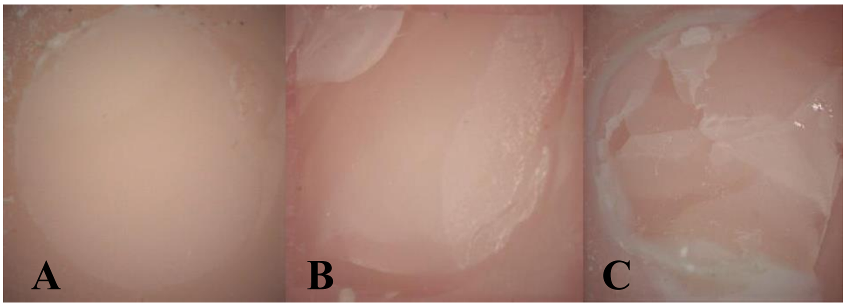

Figure 16. Failure modes under the microscope: A- Adhesive type, B- Cohesive type, C-Mixed type.

CHAPTER 6

Discussion

This study investigated the *in vitro* failure mode and shear bond strength of three commercially available resin-based cements applied to CAD/CAM nano-ceramic material. Results differed according to the cement. The light-cured adhesive resin cement presented the greatest average shear bond strength, followed by self-adhesive, and adhesive dual-cure cement. The majority of failures were caused by adhesive collapse, followed by cohesive, and mixed failure.

Currently, there is no consensus on which type of resin cement is superior to nano-ceramic CAD/CAM materials. While adhesive cements have been successfully used in clinical practice for several years, self-adhesive cements were later introduced to improve the workflow and reduce chair time, considering the reduced number of steps involved as the tooth surface does not require pre-treatment. (Makkar & Malhotra 2013) The simplicity of the luting process for self-adhesive cement reduces the risk for technique-related errors. The acidic chemical groups in self-adhesive cements etch the dental tissues and polymerization results in cross-linking of the monomers, which results in dramatic elevation of the initially acidic pH. (Radovic et al. 2008)

In the study from Alp et al. (2018), shear bond strength for Cerasmart blocks subjected to airborne-particle abrasion was ap. 10 MPa for light-cure resin cement and ap. 8.5 MPa for dual-cure resin cement. Results from the present study are consistent with the results from Alp et al. (2018), where the light-curing cement protocol presented peak shear bond strength (13.87 MPa) in comparison to the dually cured cement (9.42 MPa).

However, there is conflicting evidence on the efficacy and predictability of self-adhesive cements. (De Munck et al. 2004, Abo-Hamar et al. 2005, Goracci et al. 2006, Al-Assaf et al.

2007, Hikita et al. 2007) Considering that the type of CAD/CAM material can also influence adhesion, and the different types of material commercially available, it becomes even more difficult to conclude as to which cement is best suited for a specific material. (Kurtulmus-Yilmaz et al. 2019)

Using a similar study design, Kim et al. (2016) compared shear bond strength for a resin nano ceramic (Lava^{TM} Ultimate) when used with a light-cure adhesive cement (RelyX Ultimate clicker) and two self-adhesive cements. Similarly to the current study, the authors observed the greatest bond strength for light-cured cement (10.7 MPa) in comparison to the self-adhesive groups (≤8.9 MPa). Self-adhesive resin cements adhesive bond depends on chemical reactions with dental hydroxyapatite and micromechanical retentive factors. (Radovic et al. 2008) Thus, it can be speculated if the observed results for the light-cured adhesive cement in the present study are related to the absence of chemical reaction between hydroxyapatite and the self-adhesive cement, given that CAD/CAM slices were cemented together. In other words, the lack of dental tissues in the current *in vitro* study might have affected the observed weaker bond of the self-adhesive cement.

While the exact reason why the light-cured adhesive cement presented better bond strength is unknown. Dually cured resin cements might be influenced by the hand mixing process, which can lead to the formation of pores and voids, potentially leading to lower bond strength. (De Souza et al. 2015). According to Husain et al. (2020), clinically, dual curing cements are preferred for CAD/CAM inlays made from ceramic and resin-matrix ceramic, given that the chemical process compensates for the limited access of light in the bottom part of the cavity. Thus, the results from the current study need to be further evaluated in extracted teeth and clinical studies, since the differences between *in vitro* and clinical conditions can influence the performance of the cement.

Another factor to the taken into consideration is the lack of silane application in both adhesive cement groups. Although particle abrasion has been suggested as the most adequate surface treatment for dispersed filler CAD/CAM materials such as Cerasmart (Spitznagel et al. 2014), the use of silane coupling agents have also been suggested after particle abrasion. (Eldafrawy et al. 2019) Hence, the lack of silane might have played a role in the current results.

The majority of failures in this study were located along between the CAD/CAM material and the cement (adhesive failure mode). Toledano et al. (2007) has suggested that, in a clinical scenario, cohesive and mixed failure modes are beneficial, as they are linked to higher bond strength. In terms of failure mode, the frequency of cohesive failures ranged from 30-35% in all the groups, despite the greater bond strength associated with the light-cured group. The dual-cured adhesive cement group presented the highest frequency of mixed failures despite the lowest shear bond strength. These results suggest that shear bond strength was not related to the failure mode.

Because great part of the previously reported clinical failures of CAD/CAM restorations has been attributed to debonding (Zimmerman et al. 2018, Oz et al. 2020), it is clinically relevant to evaluate the bonding strength of CAD/CAM materials and the factors that can increase the bonding strength. Thus, further studies on different CAD/CAM materials, the influence of cementation protocols, surface treatment, the chemical composition of the cement, and curing mode.

Limitations of the study

Despite the use of a validated method that has been used in numerous studies, extrapolation of the *in vitro* results from the present study is not possible. In addition, the bonding strength of the cement to the dental structures was not evaluated. Hence, this study does not provide information on the adhesion between cement, dentin, and enamel, nor on the potential effect of the hybrid layer. The adhesive light-cured cement presented the best results *in vitro* regarding shear bond strength, however, light-curing might not be ideal in thick restorations as the light might not adequately reach the cement, as indicated in previous studies.

CHAPTER 7

Conclusions

Based on findings from this study, the following conclusions can be drawn:

- The type of resin-based cement affected shear bond strength values.
- The greatest shear bond strength was observed for the adhesive light-cured cement group, followed by the self-adhesive, and the dually cured adhesive cement.
- The adhesive failure mode was predominant in all three groups and had the highest frequency in the light-cured adhesive cement. The cohesive failure had a similar frequency in the three groups, with the self-adhesive cement group presenting the highest frequency. Failure mode did not reflect shear bond strength.
- Further research is required for additional investigation on the effect of curing mode, silane application, and the interaction between dentine and enamel on the bonding of self-adhesive cements used for CAD/CAM resin nanoceramics.

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