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BASIC RESEARCH

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Microtensile Bond Strength Between Self-Adhesive Resin Cements and Resin Based Ceramic CAD/CAM Block

Resistencia adhesiva microtensil entre cementos de resina autoadhesivos y bloques de cerámica CAD/CAM a base de resina

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ABSTRACT: Purpose: The objective of this study is to evaluate and compare the microtensile bond strength (µTBS) of four different self-adhesive resin cements to a resin-based ceramic CAD/CAM block, at the baseline, and after subjecting them to 5,000 thermo-cycles.Materials and Methods: Four self-adhesive dual-cured resin cements; G-CEM LinkAce (GC EUROPE, Leuven, Belgium), RelyX U200 (3M ESPE, Seefeld, Germany), Maxcem Elite (Kerr, CA, USA), TheraCem (Bisco, Schaumburg, USA) were applied to Cerasmart CAD/CAM blocks (GC EUROPE, Leuven, Belgium). CAD/CAM blocks were sectioned into sticks and subjected to µTBS tests at 24 hours, and the other half were subjected to tests after 5000 thermo-cycles. The data were tested by one-way variance analysis ($p<0.05$). Results: The highest bond strength values were observed in TheraCem, followed by G-CEM LinkAce and RelyX U200, respectively ($p<0.05$). At the baseline, G-CEM LinkAce, RelyX U200, and Maxcem Elite showed statistically similar results. After 5,000 thermal-cycles, a significant decrease was observed in the bond strength values of G-CEM LinkAce ($p<0.05$). Conclusion: Between the adhesive cements used in the study, TheraCem showed the highest micro-tensile bond strength values both in the baseline (24h) results, and after the 5,000 thermal-cycle aging procedures.

KEYWORDS: CAD/CAM; Resin-ceramic; Microtensile; Adhesives; Resin cements; Dental bonding.

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RESUMEN: Propósito: Evaluar y comparar la resistencia adhesiva microtensil (µTBS) de cuatro cementos de resina autoadhesivos diferentes con un bloque CAD / CAM de cerámica a base de resina, antes y después de 5.000 ciclos de termociclado. Materiales y métodos: cuatro cementos de resina de doble curado autoadhesivos; G-CEM LinkAce (GC EUROPE, Lovaina, Bélgica), RelyX U200 (3M ESPE, Seefeld, Alemania), Maxcem Elite (Kerr, CA, EE. UU.), TheraCem (Bisco, Schaumburg, EE. UU.) fueron aplicados a bloques Cerasmart CAD/CAM (GC EUROPE, Lovaina, Bélgica). Los bloques CAD/CAM se seccionaron en barras y se sometieron a pruebas µTBS a las 24 horas, y la otra mitad se sometió a pruebas después de 5000 ciclos térmicos. Los datos fueron probados por análisis de varianza unidireccional ($p<0.05$). Resultados: Los valores más altos de fuerza de unión se observaron en TheraCem, seguido de G-CEM LinkAce y RelyX U200, respectivamente ($p<0.05$). En la línea de base, G-CEM LinkAce, RelyX U200, Maxcem Elite mostró resultados estadísticamente similares. Después de 5.000 procesos de ciclo térmico, se observó una disminución significativa en los valores de resistencia de la unión de G-CEM LinkAce ($p<0.05$). Conclusión: entre los cementos adhesivos utilizados en el estudio, TheraCem mostró los valores más altos de resistencia de la unión micro-extensible tanto en la línea de base (24 h) como después de 5,000 procedimientos de envejecimiento térmico.

PALABRAS CLAVE: CAD/CAM; Resina-cerámica; Microtensil; Fuerza de Tensión; Adhesivos; Cementos de resina; Adhesión dental.

INTRODUCTION

Long-term success in ceramic or composite based indirect dental restorations depends on the luting cement selection and application procedures. Luting cement fills the interface between the restoration and prepared dental tissues and provides retention during oral functions. Properties such as adhesion strength, solubility, wettability, color harmony with ceramic or composite materials, and biocompatibility have a significant role in the proper selection of luting material (1-3).

The disadvantages of conventional cements such as higher solubility, poor adhesion, and pulpal irritation, have led to the development of resin composite cements (1). These luting materials require adhesive systems for bonding to the dental tissue and restorative materials. Especially three-step adhesive systems are the gold standard for the optimal adhesion performance and marginal adaptation between the tooth and indirect restorative materials (4). However, these adhesive procedures are more complex, time-consuming, and technically sensitive for the practitioners. Therefore, self-adhesive resin cements (SARC) which do not need any pretreatment on dental tissues, have been developed and have become popular because it simplifies the luting process (4,5).

SARC components are functional acidic monomers such as carboxylic acid groups (e.g., 4-Methacryloyloxyethyl trimellitic acid (4-MET)) or phosphoric acid groups (e.g., 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP)), aromatic or aliphatic dimethacrylate monomers (e.g., Bisphenol A diglycidyl methacrylate (bis-GMA)), filler particles and initiator systems (5,6). Dimethacrylate monomers form a crosslinked network. Acidic monomer groups demineralize enamel and dentin, provide adhesion to the tooth surface through micromechanical retention and copolymerize with the crosslinking monomers (7). SARC initially can be highly acidic, and hydrophilic for bonding as well as for adaptation to the tooth surface, by providing demineralization (8). While the chemical reaction of acidic monomers with fillers and apatite in the tooth structure continues, pH gradually increases; and after 24 hours, the structure becomes neutral and hydrophobic. Neutralization provides high mechanical strength, low water absorption, and minimum hydrolytic degradation (5,6,9).

In recent years, the use of computer-aided design (CAD) and computer-aided manufacturing (CAM) systems has increased, which have no need for traditional laboratory procedures and have the advantage of preparing in a single clinical session in indirect restorations (10). Resin matrix ceramics, silicate ceramics, and oxide ceramics are used with CAD/CAM systems for indirect restorations (11,12). Resin matrix ceramics are divided into resin-based ceramics and hybrid ceramics (12). Although ceramic materials have superior mechanical, optical, and aesthetic properties, resin-based ceramics have similar flexural, abrasion, and compression characteristics as natural tooth (13,14). These materials are favorable for inlay, onlay, and veneer restorations because of their advantages, such as easier repairing and being more fracture-resistant than ceramic CAD/CAM materials (14,15).

Adhesion protocol is the key difference between hybrid and resin-based ceramics. Due to its ceramic structure, hybrid ceramics ideally require hydrofluoric (HF) acid etching and silane application for 60 seconds. In contrast, resin-based ceramics are not etched; instead, aluminum oxide sandblasting should be conducted as pretreatment and then silanized (16). Silane coupling agent, which creates a chemical bond between the CAD/CAM resin blocks and resin cements, has bifunctional characteristics promoting chemical interaction between the inorganic components of the hybrid composite blocks and the methacrylate groups of resins through siloxane bonds (17). The ratio of hydrophilic acid functional groups to hydrophobic methacrylate groups in SARC plays an important role in water absorption (18). However, there is limited specific data about the bond strength and degradation of self-adhesive resin cements with different monomer compositions. Thus, this study has aimed to compare the microtensile bond strength of four different dual-cure self-adhesive resin cement after thermocycling to a resin-based ceramic CAD/CAM block. The null hypothesis was that the cement type had no significant influence on the microtensile bond strength of CAD/CAM resin block, and bond strength values significantly decreased following thermocycling aging.

MATERIALS AND METHODS

Four self-adhesive dual-cured resin cements; G-CEM LinkAce (GC EUROPE, Leuven, Belgium), RelyX U200 (3M ESPE, Seefeld, Germany), Maxcem Elite (Kerr, CA, USA), TheraCem (Bisco, Schaumburg, USA) and one brand of resin-based ceramic CAD/CAM block Cerasmart (GC EUROPE, Leuven, Belgium) were used in this study. Brands and compositions of the materials are shown in Table 1.

Table 1. The Composition of the Tested Materials.

Material	Type	Manufacturer	Composition
Cerasmart	Resin Based Nanoceramic CAD-CAM Block	GC Corp., Tokyo, Japan	Nanoparticle-filled resin 71 wt% silica and barium glass fill, 2,2-bis(4-methacryloxpolyethoxyphenyl) propane (Bis-MEPP), Urethane dimethacrylate (UDMA), dimetacrylate (DMA).
G-CEM LinkAce	Dual-cure Self-adhesive Cement Auto Mix System	GC EUROPE, Leuven, Belgium	UDMA, dimethacrylate, phosphonate monomer, γ-methacryloxypropyltrimethoxysilane, α,α-dimethylbenzyl hydroperoxide, fluoro alumino silicateglass, silicon dioxide, initiator, inhibitor, pigment.
RelyX U200	Dual-cure Self-adhesive Cement	3M ESPE, Seefeld, Germany	Base paste: Methacrylate monomers containing phosphoric acid groups, Methacrylate monomers, Silanated fillers, Initiator components, Stabilizers, Rheological additives Catalyst paste: Methacrylate monomers, Alkaline(basic) fillers, Silanated fillers, Initiator components, Stabilizers, Pigments, Rheological additives.
Maxcem Elite	Dual-cure Self-adhesive Cement	Kerr, CA, U.S.A.	Glycerol phosphate dimethacryalte (GPDM), Co-monomers, Proprietary self-curing redox activator, Camphorquinone, Stabilizer, Barium glass fillers, Fluoroalumino silicate glass filler, silica.
TheraCem	Dual-cure Self-adhesive Cement	Bisco, Schaumburg, U.S.A.	Base: calcium base filler, glass filler, dimethacrylates, ytterbium fluoride, initiator, amorphous silica Catalyst: glass filler, Methacryloyloxydecyl Dihydrogen Phosphate (MDP), amorphous silica.
G-Multi PRIMER II	Ceramic Primer	GC Corp., Tokyo, Japan	Ethanol, Methacryloyloxydecyl Dihydrogen Phosphate (MDP), Methacryloyloxydecyl dihydrogen thiophosphate (MDTP), γ-Methacryloxypropyl trimethoxysilane (Silane), Methacrylate monomer Application protocol: Apply a thin layer to the adherent surface of the restoration using a micro-tip applicator and dry with an air syringe.

Four CAD/CAM blocks were used for each self-adhesive resin cement group. 4 mm of slabs were sectioned with a diamond blade using a precision cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). Then top surface polishing was performed using 600-grit SIC papers for 60 seconds under running water and then sandblasted with Al2O3 (mean particle size 50 μm) (Renfert GmbH, Hilzingen, Germany). For sandblasting, a sandblaster machine (Basic Eco, Renfert GmbH) was used at 2.5-bar pressure, approximately 10 mm from the surface for 15 seconds.

After sandblasting, for the specimen silanization, G-Multi PRIMER (GC) was used for 30 seconds and air-dried. Two pairs of 4-mm high CAD/CAM slab were bonded together by four different types of cement. The standard 1 kg pressure was applied for 120 seconds. Excess cement remnant was removed. The specimens were then polymerized for 40s from each side using a LED curing unit (Elipar S10, 3M ESPE, Seefeld, Germany).

Bonded slabs were sectioned into 1mm² ± 0.2 mm² sticks after 24 hours at 37°C water storage using a low-speed cutting saw. For measuring the thickness of the adhesive interface, a digital caliper was used. Half of the specimens were subjected to microtensile bond strength tests after 24 hours storage, and the other half were subjected to tests after 5000 thermo-cycles between 5 - 55°C, with 30 seconds dwell time.

Micro Tensile Tester Machine (Bisco Inc., Schaumburg, IL, USA) was placed with the control panel facing the user. The beams were attached to a stainless-steel jig using a cyanoacrylate adhesive. The tensile load was applied at a 0.5 mm/min crosshead speed until specimen failure. The values in Newton were converted to Megapascal (MPa) based on the cross-sectional area of each specimen. The failure modes were evaluated at 40x magnification using a stereomicroscope (Olympus SZ61, Munster, Germany), and they were classified as a cohesive failure within the resin block and adhesive failure at the interface. Pretest failures and cohesive failures were excluded from statistical analyses.

For the statistical analyses, SPSS for Windows version 20.0 (SPSS, Chicago, IL, USA) was used. One-way variance analysis (ANOVA), and the Tukey HSD post hoc test was used to analyze the µTBS tensile strength mean values ($p<0.05$).

RESULTS

One-way ANOVA showed that the type of self-adhesive resin cement significantly affected the µTBS values ($p=0.000$). Microtensile Bond Strength values and the percentage of adhesive failures were shown in Figure 1. In baseline, the highest bond strength values were observed in TheraCem (55.14 MPa), and it was statistically significant as compared to other self-adhesive resin cements ($p<0.05$). G-CEM LinkAce (46.84 MPa), RelyX U200 (45.28 MPa), Maxcem Elite (40.05 MPa) showed similar µTBS values at the initial period. Tukey's multiple comparison test showed no statistical difference between G-CEM LinkAce and RelyX U200 ($p=0.990$), and between G-CEM LinkAce and Maxcem Elite ($p=1.000$).

After 5,000 thermal-cycles, the highest bond strength values were observed in TheraCem (47.85 MPa), followed by Maxcem Elite (42.04 MPa), and RelyX U200 (41.67 MPa). There was a significant difference observed between G-CEM LinkAce and TheraCem ($p=0.000$).

After aging, the µTBS values of each group decreased except Maxcem Elite, but a statistically significant decrease was only observed in the bond strength values of G-CEM LinkAce ($p=0.018$).

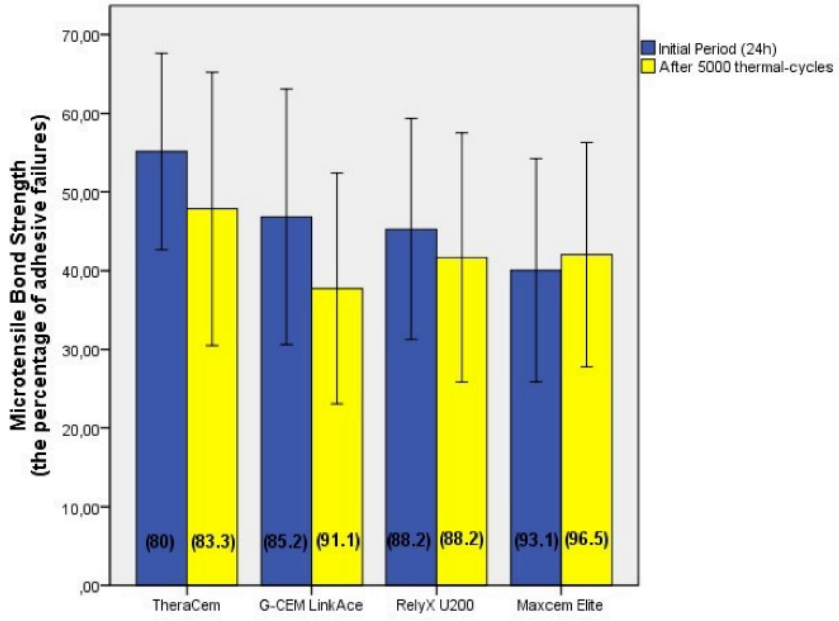

Figure 1: Mean Values ± Standard Deviation of Microtensile Bond Strength in MPa and (the percentage of adhesive failures).

DISCUSSION

Bonding effectiveness of adhesive resin cements to tooth structure has been well documented; however, there aren't enough studies about the bond strength of resin cements, especially self-adhesives to CAD/CAM materials. So, in order to address this deficit in the literature, this study has investigated the microtensile bond strengths of different self-adhesive resin cements to a resin-based ceramic CAD/CAM block. The results showed that the µTBS values were significantly influenced by luting cement type and thermo-cycle aging. The first null hypothesis that "the cement type had no significant influence on the microtensile bond strength of CAD/CAM resin block" was rejected.

Cerasmart is a resin-based ceramic CAD/CAM material that has ceramic particles embedded in a resin matrix (19). It contains 71% Silica (20 nm) and barium glass (300 nm) particles by weight. For this block, sandblasting with 25-50µm alumina and subsequently cleaning of the surface by applying the silane coupling agent, is recommended by the manufacturer. There is a wide variety of data about the surface pretreatments such as sandblasting and silane application (20-23). According to Emsermann *et al*. (21), the application of a silane coupling agent may negatively affect the bond strength on CAD-CAM materials, especially for Cerasmart; however, Lise *et al*. (24) stated the opposite that it is compulsory to use silane after sandblasting. Also, sandblasting and silanization increased µTBS values between CAD/CAM blocks and adhesive resin cements (20,22). In the present study, we focused on the bonding effectiveness of self-adhesive resin cements, and so we decided to apply the recommendations for surface pretreatment.

Functional phosphate monomers in self-adhesive resin cements, make a chemical reaction with the ceramic particles and other inorganic fillers in CAD/CAM materials, and provides optimal bond strengths (25). In the present study, the highest µTBS values were observed in TheraCem, followed by G-CEM LinkAce and RelyX U200; Maxcem Elite showed the lowest µTBS results. This condition could be associated with different types of organic monomers contained in these cements. TheraCem contains a functional 10-MDP monomer that could be accepted as a gold standard monomer, since it contains hydrophobic long carbon chains that do not attract water and have superior hydrolytic stability according to after long-term-aging micro-tensile bond strength tests (26,27). MDP has a bifunctional adhesive monomer that can bond to zirconia, metal, or silica. The hydrophilic phosphate terminal end of MDP chemically interacts with oxides on the inner surface of the restorations, while the hydrophobic methacrylate terminal end copolymerizes the resin monomers of cement (28). G-CEM LinkAce contains phosphoric ester monomers and hydrophobic monomers. However, it's dissolution stability, and adhesion performance values are not as high as 10-MDP (29). RelyX U200 has phosphoric acid groups that form acid-base reactions with hydroxyl groups in the ceramic or other inorganic fillers of CAD/CAM materials. Its methacrylate monomers have reactive carbon double bonds that induce copolymerization with polymer material in CAD/CAM substrate (30).

According to previous studies (29,31), mechanical properties of resin cements and adhesive interface durability are considered to be proportional to the rate of inorganic filler content. In our study, the inorganic filler contents of all resin cement materials similar by weight (RelyX U200=72 wt%>G-CEM LinkAce=71.4 wt%>Maxcem Elite=66,9 wt%>TheraCem=60-65 wt%). Chen *et al*. (32) stated that TheraCem exhibited higher bond strength values than UniCem 2 and FujiCem 2, because TheraCem was more resistant to aging in an acidic environment than these two adhesive systems. It was stated that TheraCem contains calcium silicate, and when it was mixed, the reaction started with an acidic pH value of 4.0 and stabilized at pH 9.0 after 24 hours (32). It has a higher neutralization ability than others, which could explain the highest μTBS values observed in TheraCem.

When the self-adhesive resin materials are mixed, they exhibit low pH values, and acid-base-interactions increase pH values at the end of 24 hours. However, the pH degree of each material varies. In previous studies, at the end of 24 hours, there was no change in acidic pH value of Maxcem Elite (close to pH 4) (8). As reported by Ferracane *et al*. (6), due to its low pH value in the cured material, a hydrophilic character can compromise mechanical stability with excessive water absorption. When acidic monomers are not adequately neutralized, they might retain their etching potential, and by affecting the polymerization reaction, this weakens adhesion (33). Also, in another study, the Maxcem Elite's low bond strength results were associated with the low degree of monomer conversion compared to the others (34).

The second null hypothesis that "bond strength values significantly decrease following thermocycling aging" was partially accepted. After 5,000 thermal-cycling aging process, only G-Cem Link Ace μTBS values were significantly decreased. Also, μTBS value decreases were shown in TheraCem and RelyX U200 groups, and it was observed that the results were not statistically significant. In self-adhesive resin cements, acidic monomers with hydrophilic character and hydrophilic sites (hydroxyl, ethylene oxide, and urethane groups) of hydrophobic crosslinking monomers as Bis-GMA, TEGDMA, and UDMA are very prone to water sorption. Water sorption exerts the hygroscopic expansion and plasticization, and it leads to contraction stress. It creates microcracks ve hydrolytic degradation (35,36).

In contrast to our study, in one study, the effect of thermal aging was observed; and the bond strength of the other cements decreased, while the results of RelyX U200 increased.

It was associated with RelyX U200, as it has a moisture tolerance structure and complete polymerization of the chemically cured part during thermocycling (37). Similar to our study, Zorzin *et al*. (38) found that the μTBS value of Maxcem Elite increased after thermal cycling. Maxcem Elite consists of crosslinked monomers such as GDM, UDMA, TEGDMA, and other methacrylate monomers such as GPDM, HEMA. TEGDMA includes water-soluble hydrophilic ether linkages and HEMA, GDM and GDM were the highest water solubility monomers. Solubility leading to the expansion of the polymer matrix and creates microcracks in the adhesive interface (39).

The majority of the failure modes were adhesive. Similar to the study by Tekçe *et al*. (30), this finding is related to the value of the μTBS.

CONCLUSIONS

Within the limitations of this in vitro study, it can be concluded that self-adhesive resin cements are effectively bonded to resin-based ceramic CAD/CAM material; however, the concentration and type of phosphate monomer, and other monomer contents in self-adhesive resin cements had a significant effect on the bond strength. Additional clinical research and in vitro studies may be necessary for verifying these results.

CONFLICTS OF INTEREST

None.

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