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Shear bond strength of self-adhesive resin cement to 3D-milled resin treated with universal adhesive

Resistência de união ao cisalhamento de cimento resinoso autoadesivo à resina composta CAD-CAM fresada tratada com adesivo universal

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ABSTRACT

Objective: To investigate the influence of 3D-milled composite resin surface pre-treatment with universal adhesive to shear bond strength (SBS) of self-adhesive resin cement. **Material and Methods:** 45 samples from hybrid CAD-CAM resin blocks (Brilliant Crios, Coltene, Altstätten, Switzerland) were cut with 15 × 10 × 1.5 mm and divided into 3 groups (n=15) according to resin surface pre-treatment: OC group – One Coat 7.0 universal adhesive and SoloCem self-adhesive resin cement (Coltene, Altstätten, Switzerland); OCA group – One Coat 7.0 chemically activated and SoloCem; SC group (control) – SoloCem, without resin surface pre-treatment. The samples of each group were divided into 3 subgroups (n=5) according to period of aging in distilled water at 37 °C until theSBS test: 1h (baseline); 7 days; and 14 days. SBS test was performed by universal testing machine (5566A, Instron; Massachusetts, USA), operating with 0.5 mm/min speed and 1k-N weight. A stereomicroscope was used to investigate failure pattern. The statistical analysis was performed with two-way ANOVA followed by Tukey tests ($\alpha = 0.05$). **Results:** OC and OCA groups showed greater SBS than SC-control group ($p < 0.05$) in all periods analysed. SC group showed a significantly SBS increase after 7 days which remained unchanged after 14 days. Adhesive failures were associated with SC group, and some mixed failures with OC and OCA groups after 7 e 14 days. **Conclusion:** The 3D-milled composite resin surface pre-treatment with an adhesive allowed best SBS of self-adhesive resin cement.

KEYWORDS

Composite resins; Computer-aided design; Dental cements; Dentin-bonding agents; Shear strength.

RESUMO

Objetivo: Investigar a influência do tratamento de superfície com adesivo universal de uma resina composta CAD-CAM fresada sobre a resistência de união ao cisalhamento (SBS) do cimento resinoso auto-adesivo. **Material e Métodos:** 45 amostras de blocos híbridos de resina CAD-CAM foram seccionadas contendo 15 mm × 10 mm × 1.5 mm e divididas em 3 grupos (n=15), de acordo com os seguintes pré-tratamentos: grupo OC – adesivo universal OneCoat 7.0 e cimento resinoso auto-adesivo SoloCem; grupo OCA – OneCoat 7.0 quimicamente ativado e SoloCem; grupo SC (controle) – SoloCem sem pré-tratamento da superfície da resina. As amostras de cada grupo foram divididas em 3 subgrupos (n=5), de acordo com o período de envelhecimento em água destilada, a 37 °C: 1h (imediato); 7 dias e 14 dias. O teste SBS foi realizado por meio de máquina de ensaio universal, operando com 0,5 mm/min e 1k-N. Uma lupa estereomicroscópica foi usada para investigar o padrão de falhas. Análise estatística foi realizada utilizando-se ANOVA e teste Tukey ($\alpha = 0{,}05$). **Resultados:** Os grupos OC e OCA apresentaram maior SBS comparados ao SC-controle ($p < 0{,}05$) em todos os períodos. O grupo SC mostrou um aumento significativo da SBS após 7 dias, a qual permaneceu inalterada aos 14 dias. Falhas adesivas foram associadas com o grupo SC e algumas falhas mistas com os grupos OC e OCA, após 7 e 14 dias. **Conclusão:** O pré-tratamento de superfície da resina composta CAD-CAM fresada com um adesivo universal permitiu maior resistência de união ao cimento resinoso auto-adesivo.

PALAVRAS-CHAVE

Resinas compostas; Desenho assistido por computador; Cimentos dentários; Adesivos dentinários; Resistência ao cisalhamento.

INTRODUCTION

Computer-aided design/computer-aided manufacturing (CAD-CAM) technology has allowed digital planning and production of indirect dental restorations with different restorative dental materials, contributing to increasing the versatility and precision of oral rehabilitations and reducing time and the cost of creating restorations [1,2]. In recent decades, the continuous improvement of composite resins, dental ceramics and adhesive dentistry has contributed to the development of a new group of CAD-CAM restorative materials, which were developed to combine the advantages of polymers with the superior aesthetics of ceramics, being classified as “Resin-Matrix-Ceramics” (RMCs). These materials are also known as “hybrid ceramics” or “nanoceramics” [3,4]. Hybrid CAD-CAM materials are clinically indicated for adhesive restorations inlays, onlays, veneers and full crowns. These materials are characterized as resin-based composites (RBCs), consisting of a pre-polymerized composite resin with high percentage by weight of dispersed inorganic filler particles (60-86%, approximately) and an organic phase (14-40% by weight, approximately) [4-9].

When comparing milled indirect restorations made from ceramic CAD-CAM blocks with those from hybrid blocks, we can bring together the following advantages associated with the latter: lower technical sensitivity [5], possibility of performing intraoral repairs [2], and hardness and elastic modulus closer to those of dentin, which facilitates the milling and finishing processes of these materials when compared to glass and polycrystalline ceramics [7,10]. However, RBC is also characterized by some limitations, such as frequent short-term retention failures in the oral cavity [2,11].

The industrially standardized polymerization of these hybrid resin blocks, under high temperature, produces a material with high mechanical and physical properties, such as satisfactory color stability and flexural resistance. Although the flexural strength reported for these blocks is less than half that reported for the most commonly used ceramic blocks, they show better resistance to fatigue loading, as well as less surface wear [1,12,13]. On the other hand, the high temperature used in the manufacturing process of RBCs results in a high degree of conversion of its monomers and a low concentration of C=C bonds available on the surface of the material for an effective union with different adhesion systems. Thus, the main limiting factor is the low bond strength between RBCs and resin cementing agents [14].

A reliable bond between the dental substrates and the inner surface of the restoration is of crucial importance for the long-term clinical success rate of adhesive restorations [13,15]. Resin cements are commonly composed of dimethacrylate monomers, inorganic filler particles and an activator-initiator system for the polymerization reaction. Regarding the joining strategy, these cements can be classified as conventional, which require the prior use of a self-etching or etch-and-rinse adhesive system, and self-adhesive, which save this surface pre-treatment phase by adding functional monomers, such as 10-methacryloyloxidecyl dihydrogen phosphate (MDP) and 4-methacryloxyethyl trimellitic anhydride (4-META). Despite their low reactivity, these functional monomers allow chemical bonding between dental substrate and properly prepared ceramic, resinous or metallic surfaces. Furthermore, prior treatment of the internal surface of the restorative material can improve the performance of self-adhesive resin cements [2,10,16,17].

The lack of evidence for a better understanding of the interaction of RBCs with self-adhesive resin cements has led different researchers to investigate possible adhesion protocols to improve the clinical longevity of restorations made with these materials [7]. There is much controversy about the influence of prior application of an adhesive to the internal surface of metal-free indirect restorations associated with increasing bond strength to resin cements. Some research has compared the effectiveness of the silane present in the composition of universal adhesives with that of silanes applied alone to the internal surface of dental composites and ceramics [11-13,18]. Currently, no consistent results have been obtained on the effectiveness of applying an adhesive layer as a pretreatment to the surface of composite resin-based hybrid CAD-CAM materials on bond strength with self-adhesive cementing agents.

Hence, the aim of this study was to investigate the influence of 3D-milled composite resin surface pre-treatment with light-cured and chemically activated universal adhesive to shear bond strength (SBS) of self-adhesive resin cement. The null hypothesis is the application of the universal adhesive on the 3D-milled resin surface doesn't affect the shear bond strength of self-adhesive resin cement.

MATERIAL AND METHODS

The materials used in this study and their composition descriptions are presented in Table I.

Microstructural characterization using ATR-FTIR

The characteristics of the chemical bonds that make up the 3D-milled composite resin blocks were evaluated using the FT-IR Spectrometer Frontier (Perkin Elmer; Waltham, MA, USA), equipped with an attenuated total reflectance crystal - ATR (MIRacle ATR; Pike Technologies; Madison, WI, USA), using 32 scans at a resolution of 4 cm^{-1}. The sample was evaluated in the wavelength range between 4000-500 cm^{-1}. The spectra obtained from the sample surfaces were reported as absorbance values in percentage (%) as a function of wave number.

Shear bond strength (SBS)

A total of 45 plates measuring 15 × 10 × 1.5 mm were obtained by sectioning 3 blocks of CAD-CAM Brilliant Crios composite resin, A3-HT (Coltene, Altstätten, Switzerland) with the aid of a diamond disc (Isomet 1000; Buehler, Lake Bluff, IL). The resin plates were randomly divided into 3 groups (n = 15), according to the treatment previously applied to the resin surface for their adhesion to the SoloCem self-adhesive resin cement (Coltene, Altstätten, Switzerland): group OC - adhesive system universal One Coat®7.0 light-cured (Coltene, Altstätten, Switzerland); OCA group - chemically activated One Coat®7.0 universal adhesive system (Coltene, Altstätten, Switzerland); SC - control group, without application of adhesive prior to SoloCem cement.

Pre-treatments were carried out following the manufacturer's instructions: OC group (n = 15) - One Coat® 7.0 was applied in a thin layer for 20s on the surface of the 3D-milled resin and gently dried for 5s using oil-free compressed air before applying the SoloCem cement.OCA group (n = 15) - one drop of One Coat® 7.0 was mixed with its chemical activator One Coat®7.0 Activator for 5 to 10 s. This chemically activated adhesive was applied with a microbrush as a thin layer for 20s onto the 3D-milled resin surface and gently dried for 5s using oil-free compressed air, prior to application of SoloCem cement. SC-control group (n = 15) - Solo Cem self-adhesive resin cement was applied directly to the surface of the 3D-milled resin without any additional prior treatment.

Table I - Description of the materials used in the study

Material	Manufacturer	Composition[1]
BRILLIANT Crios Block	Coltene	Bis-MEPP, UDMA, DMA, amorphous SiO_2 (< 20nm), barium glass (< 1nm)
OneCoat 7.0 Universal	Coltene	HEMA, UDMA, 10-MDP, ethyl alcohol solution, water, photoinitiator
OneCoat 7.0 Activator	Coltene	Ethyl alcohol solution, water, chemical activator
SoloCem	Coltene	UDMA, TEGDMA, 4-META, HEMA, coatedzinc oxide, dibenzoyl peroxide, benzoyl peroxide

[1]Bis-MEPP: 2,2-bis (4-methacryloylethoxyphenyl) propane; UDMA: urethanedimethacrylate; DMA: dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-metacryloyloxydecyl dihydrogenphosphate; TEGDMA: triethyleneglycoldimethacrylate; 4-META: 4-methacryloxyethyl trimelliticanhydride.

All specimens were constructed on the surface of the 3D-milled composite resin plates using a prefabricated matrix measuring 2.3 mm in diameter and 5 mm in height (Ultradent Jig; Ultradent Products Inc., South Jordan, USA). A newly manipulated portion of SoloCem dual resin cement was inserted into the matrix using a self-mixing tip, following the manufacturer's instructions. Excess cement was removed, and the sample was light-cured for 20s (Elipar Deep Cure; 3M ESPE, Seefeld, Germany). The intensity of the light-activating LED was constantly checked with checkMarc® (BlueLight Analytics Inc., Halifax, NS, Canada). The LED had a light intensity of 1800 mW/cm^2.

After photoactivation, each group was subdivided into 3 subgroups (n = 5), according to the storage time in distilled water in a bacteriological oven at 37 °C:1 hour (baseline), 7 days and 14 days.

The SBS was tested using a universal testing machine (5566A, Instron; Massachusetts, USA) at a speed of 0.5 mm/min and a load of 1k-N. All specimens were placed in the holding device of the testing machine with the joining surface parallel to the force loading direction. The load was applied with a half-moon guillotine. The samples were loaded until failure occurred. SBS was calculated according to the following equation:

$$SBS\left[N/mm^2 = MPa\right] = F/A \qquad (1)$$

where: F = fracture load [N], A = adhesive area [mm^2]) [19].

Failure type analysis

The failure types of the fractured samples were investigated using a stereomicroscope (BX60M; Olympus Corporation, Japan) [20]. The failures observed at the interface between the resin cement and the 3D-milled resin surfaces were classified as adhesive. Failures observed in the 3D-milled resin or resin cement layer were classified as cohesive, and those observed in both areas were classified as mixed failures [19].

Statistical analysis

The SBS values in MPa were considered to describe the means and standard deviation. Two-way ANOVA and post hoc Tukey HSD test ($\alpha = 0.05$) were applied using SPSS software. Data from failure types were presented as percentage values for each failure mode.

RESULTS

ATR-FTIR

The FTIR spectra showed the following absorption bands (Figure 1): 1-4 peaks- 803 cm^{-1}, 1040 cm^{-1} and 1111 cm^{-1} due to the presence of siloxane bonds (Si – O – CH_3); 5-11 peaks- 1382 cm^{-1}, 1413 cm^{-1}, 1458 cm^{-1}, 1512 cm^{-1} and 1606 cm^{-1} due to vibration of aromatic C = C bonds; peak number 12- 1640 cm^{-1} due to vibration of the aliphatic C = C bond; peak number 13- 1726 cm^{-1} due to C=O vibration.

The high intensity of the 3-4 peaks (1040-1111cm^{-1}; Figure 1) is related to the high percentage of siloxane bonds between inorganic filler particles and resin organic matrix. The absence of a well-defined peak around wavenumber 1638-1640 cm^{-1} (aliphatic C=C bonds) indicates low concentration of available C=C bonds due to high monomer conversion quite characteristic of CAD-CAM resin blocks manufactured under high temperature and pressure using an industrial production process.

Shear bond strength (SBS)

The means and standard deviation of the SBS values (MPa) are described in Table II. ANOVA indicated a statistically significant difference between the groups. The Tukey HSD test showed higher SBS values when the 3D-milled resin surface was previously treated with universal adhesive (OC and OCA groups)

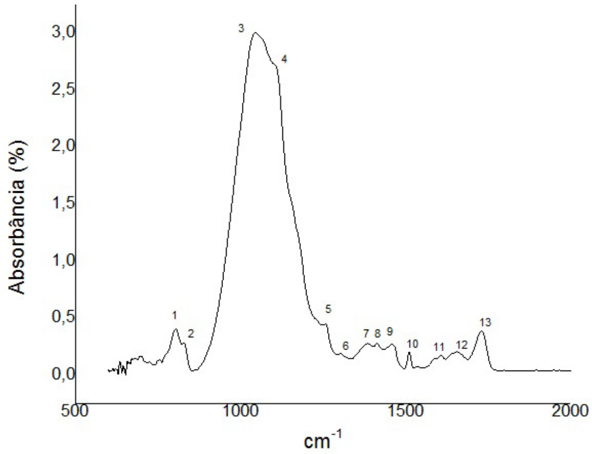

Figure 1 - ATR-FTIR spectra from 3D-milled composite resin.

compared to the untreated SC control group ($p < 0.05$), regardless of the period of analysis. There was no statistically significant difference between the different polymerization activation modes of the universal adhesive system, photo-activated (OC group) and chemically activated (OCA group), for all 3 subgroups: baseline, 7 days and 14 days ($p > 0.05$).

There was a significant increase in SBS values for the SC control group after 7 days of storage under conditions that partially simulate the oral cavity environment ($p < 0.05$), with no significant decrease after 14 days ($p > 0.05$).

Failure type analysis

The analysis of failure types for all tested specimens indicated mainly adhesive failures (Figure 2), except for the OC and OCA subgroups after 7 and 14 days of storage in distilled water, when mixed failures were observed associated with the resin cement in some specimens (Figure 3). Cohesive failures were not observed. The percentage of each failure type found is presented in Figure 4.

DISCUSSION

CAD-CAM composite resins are polymerized through an industrial process under high pressure and temperature to achieve excellent degrees of conversion of the resin monomers that make up polymeric matrix. In the current study, FTIR analyzes showed a low infrared absorbance in the wave number region around 1638-1640 cm^{-1} relative to the vibration of aliphatic C=C bonds (Figure 1). This confirms the existence of few residual resin monomers and its correlation with short SBS means and adhesive failures found mainly in the control group (SC) [21]. Such polymerization methods have resulted in high physical and mechanical properties of CAD-CAM resin blocks when compared to indirect composite resin restorations using conventional layering methods on resin dies [22]. However, when a high monomeric conversion is achieved, no bond to the composite resin can be well established without prior conditioning of its surface available for adhesion because there are no free aliphatic C = C bonds on the surface of the material, as observed in FTIR spectra (Figure 1). Due to the small number of reactive units, the adhesion of 3D-milled composite resin to resin cements appears to be difficult [2,5,14,23]. The highest SBS means observed in OC and OCA groups when compared with SC control group could show the importance that effect.

The present study revealed that prior treatment with bonding agents influences the

Table II - Description of SBS means (MPa) and standard deviation (± SD)

	Surface treatment		
	SC group (n = 15)	OC group (n = 15)	OCA group (n = 15)
Baseline (n = 5)	3.20 ± 0.23 [A, a]	14.29 ± 3.30 [A, b]	15.21 ± 2.41 [A, b]
7 days (n = 5)	9.97 ± 1.90 [B, a]	16.60 ± 4.50 [A, b]	16.66 ± 3.89 [A, b]
14 days (n = 5)	8.29 ± 4.33 [B, a]	15.70 ± 4.23 [A, b]	16.42 ± 3.83 [A, b]

Identical capital letters refer to similar subgroups in the same column. Identical lowercase letters refer to similar groups on the same line.

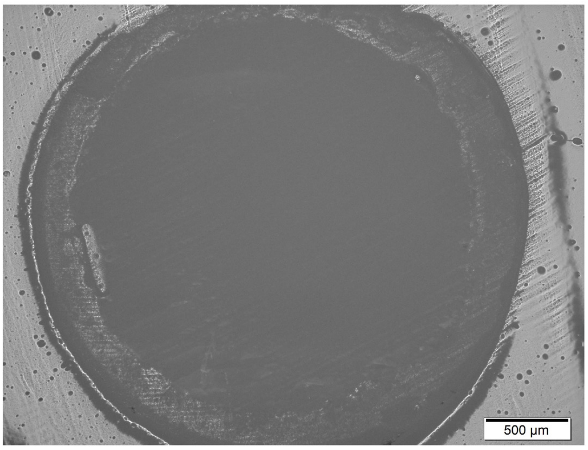

Figure 2 - Adhesive failure.

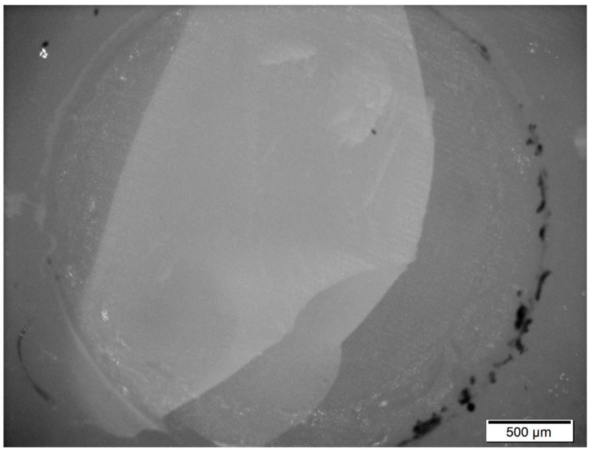

Figure 3 - Mixed failure.

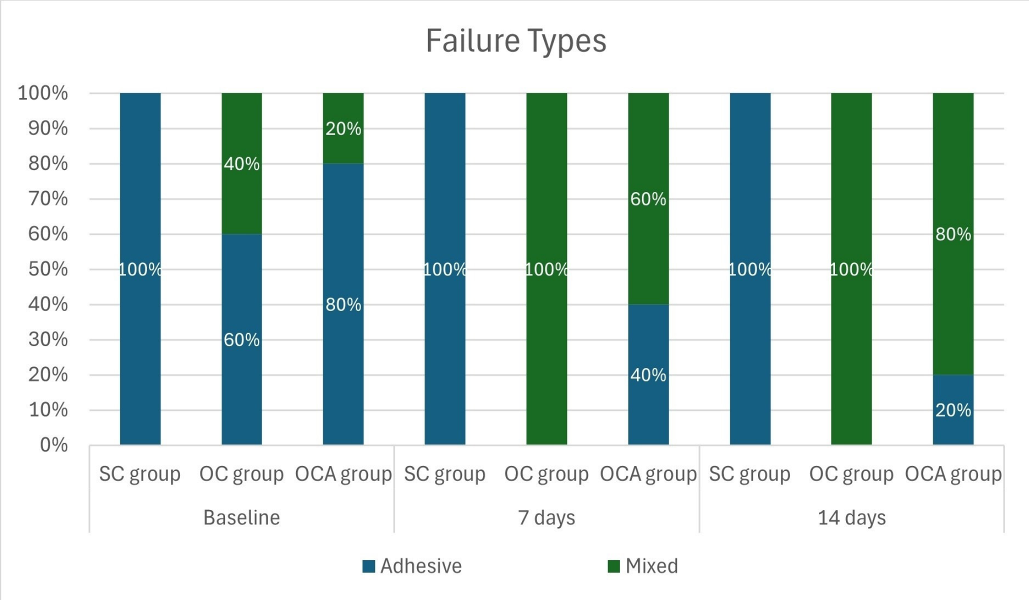

Figure 4 - Percentage of failure types.

adhesion between 3D-milled composite resin and self-adhesive resin cement. The null hypothesis was rejected. It was possible to observe the highest SBS averages when the surface of the 3D-milled composite resin was previously treated with the universal adhesive in both its activation modes, light-cured and chemically activated, when compared to the control group method, in which the cement self-adhesive resin was applied alone. This result corroborates other previous studies that tested one or more CAD-CAM composite resin blocks, using different universal adhesive systems as pre-treatment of the material surface [13,18,24].

Results for Brilliant Crios and Lava Ultimate demonstrated that the use of a universal adhesive on their internal surface prior to cementation has a favorable impact on the performance of the cementing agent [2]. Universal adhesives contain phosphate monomers and silane in their composition in addition to regular methacrylate monomers, and can outperform conventional adhesives based exclusively on regular methacrylate monomers, although this was not the subject of our current study [25].

The polymerization of the adhesives applied to the surface of the 3D-milled resin creates a strong connection between this material and the methacrylates present in resin cements, which can be called "anchoring" [26,27]. The free C = C bonds of dimethacrylates can bind to the 2 free carboxylic groups of composite resins [28]. The low pH of the universal self-etching adhesive system increases the free surface energy of the 3D-milled resin and improves the wetting capacity of the bonding agents themselves and the resin cements afterwards [2,29]. Furthermore, the inorganic filler particles in the CAD-CAM material can contribute to achieving sufficient adhesion between the 3D-milled resin and the resin cement [28].

Self-adhesive resin cements do not require prior treatment of dental hard tissues with conditioners and adhesives and can offer some advantages in clinical workflows, reducing the technical sensitivity involved in the adhesive cementation procedure in the oral cavity environment [25]. However, the presence of phosphate monomers in the composition of self-adhesive resin cements, such as the SoloCem used in this study, makes adhesion to the industrially polymerized composite resin difficult because one of the required free carbon sites remains occupied by the phosphate group [28]. Some previous studies have shown that higher bond strength can be achieved with conventional resin cements when compared with self-adhesive resin cements [14,19,28], even though such a comparison was not the aim of the current study.

A previous study comparing 3 universal adhesives as an agent for pre-treatment of the internal surface of the restorative material, Clearfil Universal Bond, Prime & Bond Universal and Single Bond Universal (SBU), demonstrated higher bond strength and predominance of cohesive failures in the groups that used the SBU [18]. The authors justify this result due to the presence of the monomer 10-methacryloxidecyl dihydrogen phosphate (MDP) in the SBU. MDP is a bifunctional monomer capable of binding to oxides and methacrylate monomers on the surface of the 3D-milledresin [18]. On the other hand, a similar study showed that the main effect of the adhesive variable was not significant, but rather the CAD-CAM block variable which means that the composition of the 3D-milledresin proved to be more relevant to the results presented by these authors than the type of joining agent used [24].

The SBS values obtained in the present study are comparable to the values presented in other studies [14,19,28]. Artificial aging in distilled water at 37 °C for 7 and 14 days exhibited a satisfactory effect on SBS for all groups, especially for the SC control group, although its high standard deviation has been considered a limitation in this study, suggesting the need for further investigations. A reason that could explain this positive effect would be the extension of the polymerization reaction of residual resin monomers for up to 21 days, especially considering a dual-curing resin cement, such as the SoloCem used in this study [21]. Previous studies have shown that the gradual increase in the degree of conversion after the initial photoactivation is related to the chemical composition of the material and not to the oral environment or laboratory study conditions [21]. It is speculated that the higher the degree of conversion of adhesives and dual resin cements applied to the cementation of CAD-CAM materials, the higher the resulting values of the adhesive mechanical properties evaluated.

Another point discussed in the present study were the types of failures, which could be adhesive, cohesive or mixed. The first one was more present in the control group, regardless of the sample storage time, and in the OC and OCA groups at baseline. Some mixed failures could be observed for the OC and OCA groups after 7 and 14 days of storage. Another study showed that all analyzed specimens exhibited complete or partial adhesive failure [13]. Adhesive failure indicates that the bond strength was lower than the cohesive forces of the materials involved. The results regarding the type of failure in the current study were similar to those of other previous studies in which adhesive failure was predominant for all groups tested, as well as for the group that did not use any surface treatment prior to the cement [15,30].

Therefore, the effectiveness of the bonding of the previous adhesive treatments was greater than the fracture resistance of the cementing agent itself considering the SBS values after 7 and 14 days of aging. Mixed failures occurred when SBS values were greater than 14.0 MPa on average. The lack of characterization of the mechanical properties of the resin cement and 3D-milled composite resin tested was a limitation in this study, suggesting the need for further investigations.

CONCLUSION

Among the limitations of this study, the prior application of the universal adhesive on the 3D-milled composite resin surface was favorable to the shear bond strength to the self-adhesive resin cement.

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Author's Contributions

LACL: Methodology, Investigation, Data Curation, Writing – Original Draft Preparation. JDM: Writing – Review & Editing. MCA: Methodology, Formal Analysis, Data Curation. HRMC: Methodology, Formal Analysis, Data Curation. CES: Conceptualization, Resources, Supervision, Project Administration.

Conflict of Interest

No conflicts of interest declared concerning the publication of this article.

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Regulatory Statement

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