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Bond strength of CAD-CAM resin composite blocks cemented with flowable or pre-heated resin composites using different adhesive strategies

Resistência de união de blocos de resina composta CAD-CAM cimentados com compósitos resinosos fluidos ou pré-aquecidos usando diferentes estratégias adesivas

Nathalie Cristina CONTENTO-FIERRO[1], Eliam Cielo ZÚÑIGA-MAYHUA[2], Jorge Alonso BORDA-BIZAGA[2], João Felipe BESEGATO[3], María Soledad PEÑAHERRERA MANOSALVAS[1], Cristian Fernando SANCHEZ-PUETATE[1], Wilfredo Gustavo ESCALANTE-OTÁROLA[2]

1 - Universidad de los Hemisferios, Facultad de Ciencias de la Salud, Posgrados Odontología. Quito, Ecuador.
2 - Universidad Católica de Santa María, Facultad de Odontología. Arequipa, Peru.
3 - Universidade Federal de Mato Grosso do Sul, Faculdade de Odontologia. Campo Grande, MS, Brazil.

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ABSTRACT

Objective: To evaluate the bond strength of CAD-CAM resin composite blocks cemented with flowable or pre-heated resin composites using different adhesive strategies. **Material and Methods:** Seventy Brava Block (FGM) CAD-CAM resin composite cylinders were milled, and one surface was treated by 50 μm alumina sandblasting, followed by ultrasonic cleaning and 70% alcohol cleaning. The cylinders were distributed into seven groups: one control group using a light-cured resin cement (Variolink Esthetic LC), three with flowable composite resin (FR), and three with preheated composite resin (PR), applying different adhesive strategies. The cementing agents were light-cured (VALO Grand, 1200 mW/cm^2) and specimens were stored at 37 °C for 24 hours. Bond strength was evaluated using shear testing (OM150, 1 mm/min), calculating average values and classifying fracture types. Data were analyzed using ANOVA and Tukey's post hoc test ($p<0.05$). **Results:** The protocol with Monobond N primer and flowable resin composite presented the highest mean bond strength values and was statistically similar to the protocols with Monobond N primer and preheated resin, and with Monobond N primer, adhesive, and flowable resin composite. However, only the later two protocols did not differ significantly from the light-cured resin cement control group. The protocol with Prosil primer and preheated resin exhibited the lowest bond strength. Mixed failures were predominant across most experimental groups. **Conclusion:** The use of Monobond N primer combined with flowable resin composite promotes favorable bond strength to CAD-CAM resin composite blocks, while pre-heated resin composites show lower performance, particularly when associated with conventional silane. The selection of the adhesive strategy plays a critical role in optimizing bonding effectiveness for these materials.

KEYWORDS

CAD-CAM systems; Composite resins; Dental adhesion; Dental materials; Silanes.

RESUMO

Objetivo: Avaliar a resistência de união de blocos de resina composta CAD-CAM cimentados com compósitos resinosos fluidos ou pré-aquecidos, utilizando diferentes estratégias adesivas. **Material e Métodos:** Setenta cilindros de resina composta CAD-CAM Brava Block (FGM) foram fresados, sendo uma das superfícies tratada por jateamento com óxido de alumínio (50 μm), seguido de limpeza ultrassônica e limpeza com álcool a 70%. Os cilindros foram distribuídos em sete grupos: um grupo controle utilizando cimento resinoso fotopolimerizável (Variolink Esthetic LC), três com compósito resinoso fluido (FR) e três com compósito resinoso pré-aquecido (PR), aplicando diferentes estratégias adesivas. Os agentes cimentantes foram fotoativados (VALO Grand, 1200 mW/cm^2) e as amostras armazenadas a 37 °C por 24 horas. A resistência de união foi avaliada por ensaio de cisalhamento (OM150, 1 mm/min), calculando-se os valores médios e classificando os tipos de fratura. Os dados foram analisados por ANOVA e teste de Tukey ($p<0,05$). **Resultados:** O protocolo com primer Monobond N e compósito resinoso fluido apresentou os maiores valores médios de resistência de união e foi estatisticamente semelhante aos protocolos com Monobond N e compósito resinoso pré-aquecido, e com Monobond N, sistema adesivo e compósito resinoso fluido. No entanto, apenas estes dois últimos protocolos não diferiram significativamente do grupo controle. O protocolo com primer Prosil e compósito resinoso pré-aquecido apresentou os menores valores de resistência de união. Falhas mistas predominaram na maioria dos grupos experimentais. **Conclusão:** O uso do primer Monobond N associado ao compósito resinoso fluido promove desempenho favorável de resistência de união em blocos de resina composta CAD-CAM, enquanto os compósitos pré-aquecidos apresentam desempenho inferior, especialmente quando associados ao silano convencional. A escolha da estratégia adesiva desempenha um papel fundamental na otimização da adesão nesses materiais.

PALAVRAS-CHAVE

CAD-CAM; Resinas compostas; Adesão dentária; Estratégias adesivas; Silanos.

INTRODUCTION

Blocks manufactured using CAD-CAM systems represent a significant advancement in dental materials, combining aesthetics, mechanical strength, and biocompatibility [1-3]. These materials allow precise fabrication of indirect restorations, improving clinical efficiency and treatment outcomes. In recent years, resin-based CAD-CAM blocks have emerged as an alternative to traditional ceramics, offering improved machinability, reduced brittleness, and simplified intraoral adjustment [4,5].

Resin-based CAD-CAM materials, also described as resin-matrix ceramics or hybrid materials, consist of an organic polymeric matrix reinforced with inorganic fillers, combining favorable mechanical properties with enhanced handling characteristics [4,6,7]. These materials exhibit adequate flexural strength, improved wear compatibility with opposing dentition, and easier polishing and repair compared to conventional ceramics [8,9]. However, their hybrid composition results in a bonding behavior that differs significantly from that of silica-based glass-ceramics.

Achieving reliable and durable adhesion to resin-based CAD-CAM materials remains challenging, as bonding depends on both micromechanical retention and chemical interaction with the organic matrix and inorganic fillers [10,11]. Additionally, these materials present a highly polymerized and relatively inert surface, which limits chemical reactivity and reduces the effectiveness of conventional silane-based approaches, making surface treatment and adhesive strategy critical factors for successful bonding [12-14].

Different cementation strategies have been proposed for resin-based CAD-CAM materials, including the use of resin cements, flowable resin composites, and pre-heated restorative composites [15]. Pre-heating composite resins has been shown to improve flowability and adaptation, although their bonding performance may vary depending on the adhesive protocol used [16,17]. However, most existing studies focus on conventional resin cements, and there is a lack of consistent evidence regarding the use of restorative resin composites as alternative luting agents for resin-based CAD-CAM materials.

Despite the growing use of resin-based CAD-CAM materials, there is still limited and inconsistent evidence regarding the use of flowable and pre-heated resin composites as alternative luting materials, particularly when combined with different silane-containing primers and adhesive systems. Therefore, this study aimed to evaluate the bond strength of CAD-CAM resin composite blocks cemented with flowable or pre-heated resin composites using different adhesive strategies. The null hypothesis was that different cementation protocols would not influence bond strength.

MATERIAL AND METHODS

Sample preparation

Seventy Brava Block (FGM; Joinville, SC, Brazil) CAD-CAM resin composite cylinders (12 mm in diameter and 14 mm in height) were fabricated using the DWX-42W CAD-CAM system (DGSHAPE Corporation; Hamamatsu, Japan). One circular surface of each cylinder was sandblasted with 50 μm alumina particles (Bio-Art; São Carlos, SP, Brazil) for 15 seconds at a pressure of 2 bar, maintaining a distance of 4 mm. The cylinders were then ultrasonically cleaned in distilled water using a CD-4820 ultrasonic bath (Codyson Ultrasonic; Shenzhen, China) for 480 seconds to remove residues, followed by cleaning with 70% alcohol.

The sample size was determined based on previous in vitro bond strength studies using similar experimental designs and was confirmed using G*Power software. Considering a one-way ANOVA fixed-effects omnibus test, seven groups, a significance level of 5%, statistical power of 80%, and a large expected effect size, a minimum of 10 specimens per group was considered adequate. Therefore, 70 specimens were included in the study.

Experimental groups

The cylinders were randomly divided into seven groups (n=10): one control group using a light-cured resin cement (Variolink Esthetic LC), and six experimental groups divided into three groups using flowable resin composite (FR) and three using pre-heated resin composite (PR), each employing different adhesive strategies. The distribution of the experimental groups is detailed in Table I.

Surface treatment and adhesive procedures

For groups treated with Monobond N, the primer was applied to the treated CAD-CAM resin composite surface for 60 s, followed by gentle air-drying to allow solvent evaporation. For groups treated with Prosil, the silane primer was similarly applied for 60 s and gently air-dried.

In groups receiving an additional adhesive system, Clearfil SE Bond was applied using its two-step protocol. The primer was actively applied for 20 s and gently air-dried, followed by application of the bonding agent, air thinning, and light-curing for 10 s using a VALO Grand light-curing unit (Ultradent; South Jordan, UT, USA) at an irradiance of 1200 mW/cm^2. After completion of the adhesive procedures, the corresponding luting material was inserted into the Tygon matrix and light-cured for 20 s using the same light-curing unit.

For the pre-heated resin groups, ENA HRI Esmalte Plus composite resin (Micerium; Genoa, Italy) was pre-heated at 55 °C for 7 minutes using a composite warmer (ENA HRI Composite Warmer, Micerium). The entire composite syringe was heated, and the material was immediately applied after removal from the device to minimize temperature loss, as pre-heated composite resins have been shown to rapidly decrease in temperature after removal from the heating device. Table II presents the specifications of the materials used.

A transparent Tygon polyethylene matrix (Anhui Hongyu Wuzhou Medical Manufacturer; Anhui, China) with an internal diameter of 2.97 mm and a height of 3 mm was employed to standardize the application of the cement, resulting in a bonding area greater than 1 mm^2, characterizing a macro shear bond strength test. The matrix was completely filled with the cementing agent to ensure homogeneous distribution, aided by a No. 5 explorer.

A cellulose strip was then placed over the matrix, and light pressure was applied to stabilize the material. Light curing was performed for 20 seconds and the specimens were subsequently stored in a humid environment at 37 °C for 24 hours. Figure 1 provides a schematic overview of the sample preparation process.

Table I - Distribution of the experimental groups

Groups		Silane-based primer	Adhesive system	Cementation agent
Control	**M-RC**	Monobond N	-	Variolink Esthetic LC
Flowable resin composite	**M-A-FR**	Monobond N	Clearfil SE Bond	ENA HRI Flow
	M-FR	Monobond N	-	
	P-FR	Prosil	-	
Pre-heated resin	**M-A-PR**	Monobond N	Clearfil SE Bond	ENA HRI Esmalte Plus
	M-PR	Monobond N	-	
	P-PR	Prosil	-	

Table II - Specifications of the materials used

Material	Type	Composition
Brava Block (FGM, Brazil, #250123-280722)	CAD-CAM resin composite block	Methacrylic resins (Bis-GMA, UDMA, TEGDMA), Ceramic particles of silica and alumina oxide, Metal oxide pigments for color and translucency, Peroxides or photoinitiator systems.
Monobond N (Ivoclar Vivadent, Liechtenstein, #Z06F4X)	Silane-based Primer	Silane, methacrylic acid, organic solvents.
Prosil (FGM, Brazil, #080422)	Silane-based Primer	Silane, organic solvents.
Clearfil SE Bond (Kuraray, Japan, #280775)	Self-etch Adhesive System	4-Meta methacrylate (4-META), Bisphenol-A glycidylmethacrylate (Bis-GMA), ethyl hydroxide, aliphatic resins, water, Silica.
Variolink Esthetic LC (Ivoclar Vivadent, Liechtenstein, #2063PL)	Photopolymerizable Resin Cement	Methacrylic resins (Bis-GMA, TEGDMA), Silica, Catalysts, photoinitiators.
ENA HRI Flow (Micerium, Italy, #2022006388)	Photopolymerizable Flowable Resin	Methacrylic resins (Bis-GMA, TEGDMA), Silicon dioxide and barium glass, Photoinitiators.
ENA HRI Enamel Plus (Micerium, Italy, #2023005422)	Photopolymerizable Composite Resin	Methacrylic resins (Bis-GMA, TEGDMA), Silicon dioxide and barium glass, Pigments.

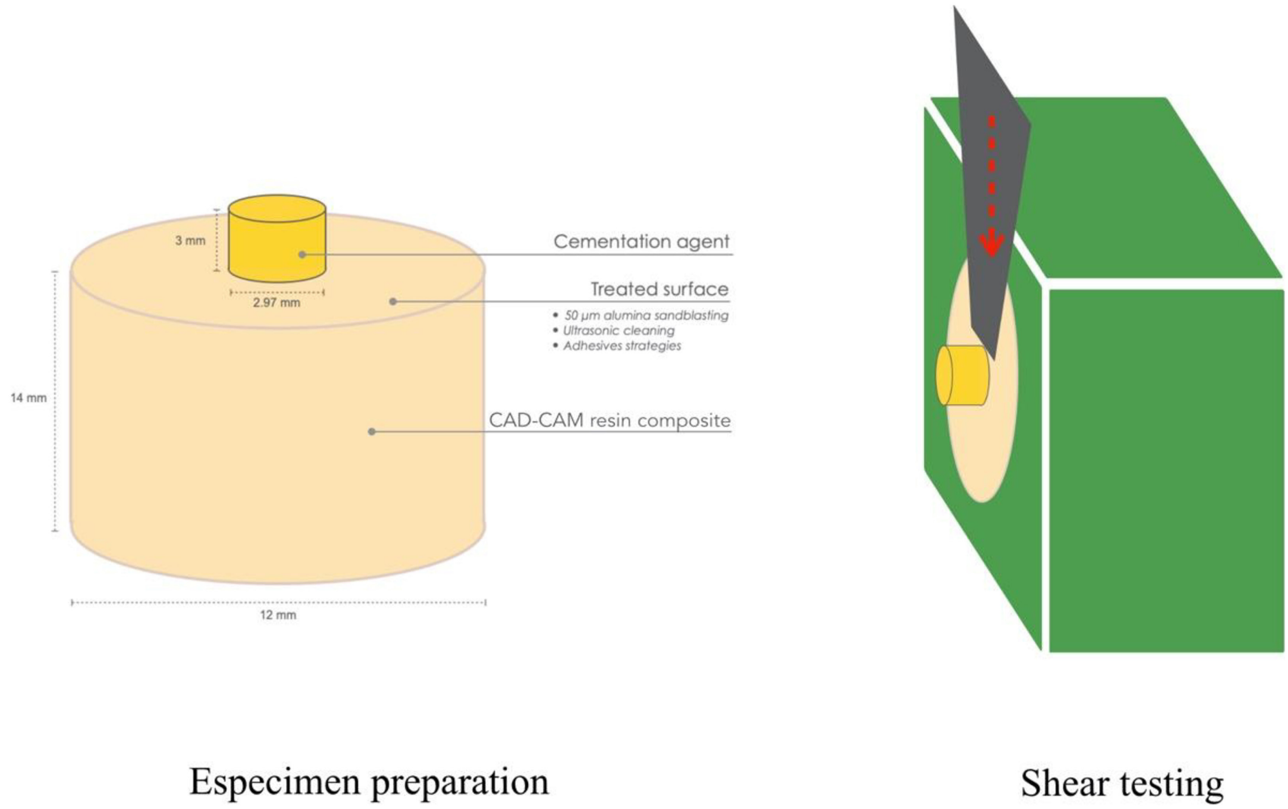

Figure 1 - Methodological scheme for the preparation of test specimens.

Bond strength analysis

After the storage period, the Tygon matrices were carefully removed using a No. 11 Biolife scalpel blade (Sterilance Medical; Suzhou, China). The specimens were examined under magnification to identify any porosities or cracks at the adhesive interface, and defective samples were discarded. No pre-test failures or specimen losses were observed during removal of the Tygon matrices.

The remaining specimens were subjected to a shear bond strength test using an OM150 universal testing machine (Odeme Dental Research; Luzerna, Brazil). Each specimen was positioned perpendicular to the load cell (1 kN) using a custom-made metallic fixture, and the test was conducted at a crosshead speed of 1 mm/min until fracture occurred. A chisel-shaped loading device was used to apply the load at the interface between the CAD-CAM block and the resin cylinder. Bond strength values were calculated by dividing the maximum load (N) by the adhesive area (mm^2), and the arithmetic mean was determined for each group [18].

Failure modes were evaluated at 10× magnification and classified into four categories: adhesive failure (AD), cohesive failure within the resin cement (CC), cohesive failure within the CAD-CAM resin composite block (CB), or mixed (M), representing a combination of fracture types.

Statistical analysis

Data normality was assessed using the Shapiro-Wilk test, and homogeneity of variances was evaluated using Levene's test. Parametric analysis was performed using one-way ANOVA followed by Tukey's post hoc test, with a significance level set at 5%. The frequency of fracture types was reported descriptively. Due to the experimental grouping and the absence of a fully balanced factorial design, comparisons were performed using one-way ANOVA to evaluate differences among the experimental conditions.

RESULTS

Table III presents the mean bond strength values, standard deviations, and confidence intervals for each cementation protocol. The M-FR group (Monobond N + flowable resin composite) presented the highest mean bond strength value (11.63 ± 0.87 MPa), followed by the M-PR (10.74 ± 0.79 MPa) and M-A-FR (10.40 ± 0.71 MPa) groups.

Table III - Mean, standard deviation, and confidence intervals of bond strength values (MPa) for the CAD-CAM resin composite blocks according to the cementation protocol used

Protocols	Control	Flowable resin composite			Preheated resin		
	M-RC	M-A-FR	M-FR	P-FR	M-A-PR	M-PR	P-PR
Mean	9.24[bc]	10.40[ab]	11.63[a]	9.37[bc]	8.73[c]	10.74[ab]	6.23[d]
SD	0.55	0.71	0.87	0.94	0.95	0.79	0.81
CI	8.90-9.58	9.96-10.84	11.10-12.17	8.79-9.95	8.14-9.32	10.25-11.22	5.73-6.74

[ab] Different lowercase letters within the same row indicate statistically significant difference (One-way ANOVA and Tukey's post-hoc test; $p < 0.05$). SD: standard deviation; CI: confidence intervals; M-RC: Monobond N and Variolink Esthetic resin cement (control); M-A-FR: Monobond N, Clearfil SE Bond adhesive, and ENA HRI Flowable resin; M-FR: Monobond N and ENA HRI Flowable resin; P-FR: Prosil and ENA HRI Flowable resin; M-A-PR: Monobond N, Clearfil SE Bond adhesive, and ENA HRI Enamel Plus preheated resin; M-PR: Monobond N and ENA HRI Enamel Plus preheated resin; P-PR: Prosil and ENA HRI Enamel Plus preheated resin.

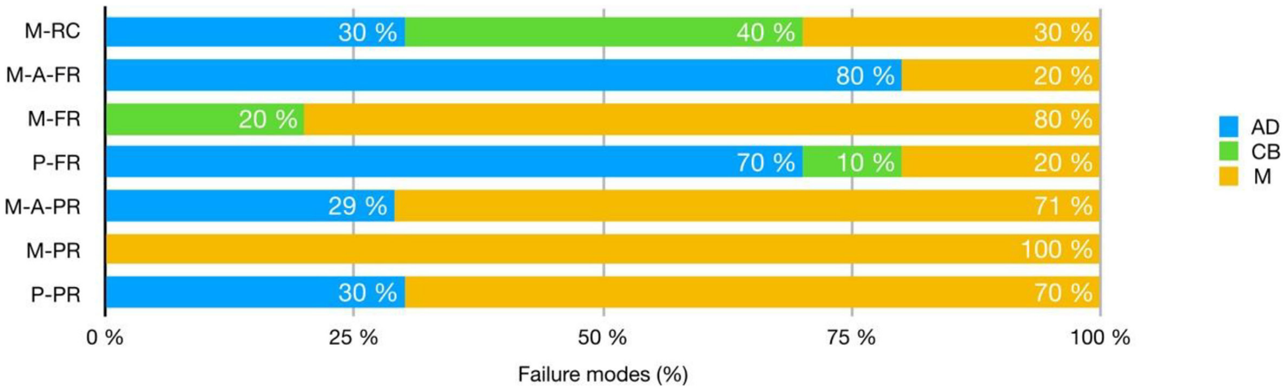

Figure 2 - Frequency distribution (%) of failure modes according to the cementation protocol. AD: adhesive; CB: cohesive within the CAD-CAM resin composite block; M: mixed failure. M-RC: Monobond N and Variolink Esthetic resin cement (control); M-A-FR: Monobond N, Clearfil SE Bond adhesive, and ENA HRI Flowable resin; M-FR: Monobond N and ENA HRI Flowable resin; P-FR: Prosil and ENA HRI Flowable resin; M-A-PR: Monobond N, Clearfil SE Bond adhesive, and ENA HRI Enamel Plus preheated resin; M-PR: Monobond N and ENA HRI Enamel Plus preheated resin; P-PR: Prosil and ENA HRI Enamel Plus preheated resin. Data are presented as percentage frequency.

However, no statistically significant differences were observed among these groups ($p > 0.05$).

Similarly, the control group (M-RC: Monobond N + Variolink Esthetic LC), the P-FR group (Prosil + flowable resin composite), and the M-A-PR group (Monobond N + adhesive system + pre-heated resin composite) showed moderate bond strength values, without significant differences among them ($p > 0.05$).

The P-PR group (Prosil + pre-heated resin composite) presented the lowest bond strength values (6.23 ± 0.81 MPa), which were significantly lower than those of all other groups ($p < 0.05$).

Figure 2 shows the distribution of failure modes for each experimental group. Mixed failures (M) were the most frequent in most groups, including M-FR, M-A-PR, M-PR, and P-PR. Adhesive failures (AD) predominated in the M-A-FR and P-FR groups. In the control group (M-RC), failures were distributed among adhesive (AD), cohesive within the CAD-CAM resin composite block (CB), and mixed (M) types, with a predominance of cohesive failures within the block. Failure modes were expressed as percentage frequency.

DISCUSSION

Our results showed that cementation protocols using flowable resin composite tended to present higher bond strength values compared to pre-heated resin composites. However, protocols based on Monobond N, including those combined with pre-heated resin, showed comparable performance, indicating that the adhesive strategy plays a key role in bonding effectiveness. The protocol using Prosil combined with pre-heated resin exhibited the lowest bond strength, leading to the rejection of the null hypothesis.

The performance of Monobond N, a silane-based primer, in enhancing bond strength may be related to its multifunctional composition, which promotes chemical interaction with both the organic matrix and inorganic fillers of resin-based CAD-CAM materials [19,20]. This is consistent with previous studies reporting improved bonding performance when multifunctional primers are used on CAD-CAM composite substrates [21]. Our findings suggest that Monobond N combined with flowable resin composite provides bonding performance comparable to other Monobond N–based protocols.

Although pre-heated resin composites have been associated with improved adaptation and handling properties [16,17], their bond strength in the present study was generally lower than that of flowable resin composites. However, when combined with Monobond N, pre-heated resin composites showed comparable performance, suggesting that the effectiveness of the adhesive protocol may compensate for differences in the luting material. This behavior may be related to the interaction between the functional components of the primer and the organic matrix of the CAD-CAM resin composite, which could enhance interfacial compatibility and bonding stability.

Previous studies have shown that the effectiveness of silane-based primers depends on their composition and interaction with the substrate [22,23]. In this context, the lack of additional benefit observed with the use of an adhesive system after silane application is consistent with reports indicating that combining silane and adhesive systems does not always improve bond strength [22].

This may explain why the inclusion of Clearfil SE Bond did not result in significant differences in the present study.

Monobond N and Prosil, while both silane-based primers, differ in formulation and reactivity. Monobond N contains functional monomers that may enhance its interaction with resin-based CAD-CAM materials [24], whereas Prosil is based on a more conventional silane composition [25]. This difference may explain the lower bond strength values observed in the Prosil groups, particularly when combined with pre-heated resin composites.

Flowable resin composites may favor better adaptation at the bonding interface due to their lower viscosity [26]. In the present study, protocols using flowable resin composites, including the control group with Variolink Esthetic LC, tended to show higher bond strength values compared to pre-heated resin composites. However, the absence of statistically significant differences among several groups suggests that viscosity alone does not determine bonding effectiveness.

The comparison between the control group and experimental protocols suggests that flowable composite resins may represent a viable alternative to conventional light-cured resin cements under certain conditions [27]. However, these differences should be interpreted with caution, as multiple factors—including adhesive strategy and substrate interaction—may influence the final bonding outcome.

Regarding failure modes, mixed failures predominated in most groups, indicating a complex interaction at the bonding interface. The presence of mixed failures even in groups with higher bond strength values suggests that bond strength alone does not fully explain the failure pattern, and that interfacial characteristics play an important role [28].

This study has limitations, including its in vitro design and the absence of aging procedures, which may influence long-term bonding performance. Additionally, only one type of CAD-CAM resin composite block and specific adhesive systems were evaluated. Future studies should include aging protocols, fatigue testing, and evaluation of different materials and adhesive strategies to better simulate clinical conditions.

Within these limitations, the results suggest that adhesive strategy plays a critical role in bonding effectiveness to CAD-CAM resin composite blocks, and that multifunctional primers such as Monobond N may provide consistent performance across different cementation approaches.

CONCLUSIONS

Within the limitations of this study, the results suggest that the selection of the adhesive strategy plays a key role in bonding effectiveness to CAD-CAM resin composite blocks. Monobond N–based protocols showed consistent performance across different luting approaches. Flowable resin composites tended to present higher bond strength values, while pre-heated resin composites showed comparable performance when combined with Monobond N. In contrast, conventional silane–based protocols were associated with lower bond strength values.

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Data availability

Data will be made available on request.

Author's Contributions

CFSP, WGEO: Conceptualization. NCCF, ECZM, JABB: Methodology. MSPM, CFSP: Software. MSPM, CFSP, WGEO: Validation. MSPM, CFSP, WGEO: Formal Analysis. NCCF, ECZM, JABB, JFB, MSPM, CFSP, WGEO: Investigation. CFSP, WGEO: Resources. NCCF, ECZM, JABB: Data Curation. NCCF, MSPM: Writing – Original Draft Preparation. JFB, CFSP, WGEO: Writing – Review & Editing. WGEO: Visualization. CFSP, WGEO: Supervision. WGEO: Project Administration.

Conflict of Interest

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

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Regulatory Statement

Not applicable.

Ethics statement

The study involved only laboratory testing of dental materials and did not require ethical approval.

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Wilfredo Gustavo Escalante Otárola
(Corresponding address)
Universidad Católica de Santa María, Facultad de Odontología, Arequipa, Peru.
Email: wilfredo.escalante@ucsm.edu.pe

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