

In vitro study of the relationship between bond strength to dental enamel and microleakage in photopolymerizable composite resins
Estudio in vitro de la relación entre resistencia de unión a esmalte dental y microfiltración en resinas compuestas fotopolimerizables
Estudio in vitro de la relación entre resistencia de unión a esmalte dental y microfiltración en resinas compuestas fotopolimerizables

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Resumen

Objetivos. Evaluar y comparar la relación entre la resistencia de unión inmediata a esmalte y microfiltración de dos sistemas restauradores a base de resina compuesta. **Métodos.** 40 terceros molares se dividieron aleatoriamente en dos grupos: 20 molares restaurados utilizando el sistema Adper Single bond 2 + Filtek Z250 XT (3M ESPE; ST PAUL, MN, USA); y 20 molares restaurados con el sistema Tetric N Bond + Tetric N Ceram (Ivoclar Vivadent; Schaan, Liechtenstein). La mitad de los dientes de cada grupo se utilizaron para un ensayo de microtracción, mientras que la otra mitad fueron utilizados para un ensayo de microfiltración (n = 10).

Resultados. Las medias y el desvío padrón de la resistencia de unión a microtracción fueron de 27.93 (± 9.55) para Adper Single bond 2 + Filtek Z250 XT MPa y 33.12 (± 8.18) MPa para Tetric N Bond + Tetric N Ceram (p = 0,049). En cuanto a los valores de microfiltración, no hubo diferencias estadísticamente significativas entre los grupos (p = 0,478). No fue observada una correlación significativa entre la resistencia de unión a la microtracción y la microfiltración ($R^2 = 0.0909$; p = 0.196).

Conclusiones. No se encontró una relación entre los valores de resistencia de unión y el grado de microtracción.

Palabras claves: Microtracción, Microfiltración, Resinas Compuestas.

Abstract

Objectives. To evaluate and compare the relationship between enamel bond strength and microleakage of two composite-based restorative systems.

Methods. Forty third molars were randomly divided into two groups: 20 molars restored with the Adper Single bond 2 + Filtek Z250 XT (3M ESPE; ST PAUL, MN, USA) system and 20 with the Tetric N Bond + Tetric N Ceram (Ivoclar Vivadent; Schaan, Liechtenstein) system. Half the teeth in each group were subjected to a bond strength test, while the others were subjected to the microleakage test.

Results. The mean and standard deviation of bond strength were 27.93 (\pm 9.55) MPa for Adper Single bond 2 + Filtek Z250 XT system and 33.12 (\pm 8.18) MPa for Tetric N Bond + Tetric N Ceram system ($p = 0.049$). Regarding microleakage values, there were no statistically significant differences between the groups ($p = 0.478$). No significant correlation was observed between microtensile bond strength and microleakage ($R^2 = 0.0909$, $p = 0.196$).

Conclusions. No relationship was found between adhesive bond values and degree of microleakage.

Keywords: bond strength; microleakage; composite resins

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Introduction

Since their introduction in the 1960s, composite resins have been increasingly used. Currently, they are considered materials for universal use and are the first option for direct restorations in anterior and posterior teeth⁽¹⁾. The aesthetic characteristics they provide, matching the natural tooth color, their ability to adhere to the hard tissues through micromechanical retention adhesive

systems, and their low cost compared to indirect restorative materials are some of the reasons that justify their use⁽²⁾.

However, a large number failures are still reported for this type of restoration in the short and medium term⁽²⁾. This is related to problems regarding the changes the resin undergoes once polymerized (polymerization contraction, thermal expansion coefficient), and regarding the oral cavity (superficial abrasion, chemical degradation, etc.)⁽³⁻⁴⁾. These problems explain restoration failures, with failures in the tooth-restoration interface, microleakage and subsequent caries recurrence⁽⁵⁻⁸⁾.

To counteract these problems, manufacturers have modified resin composition, for example, monomers with higher molecular weight (producing less polymerization shrinkage), or improving inorganic charge particles (higher load, smaller size and different morphologies), which has led to significant improvements in material properties⁽⁹⁻¹⁰⁾.

Using accelerated aging techniques under standardized laboratory conditions is an effective alternative to study long-term restorative material behavior, as it is possible to simulate the clinical use of a material in a short period. Some methods have been proposed to this end, including immersion in water, ethanol or artificial saliva, as well as thermal cycling and UV aging⁽¹¹⁾.

Thermal cycling involves subjecting samples to extreme temperatures to simulate intraoral changing temperature conditions⁽¹²⁾. The effect of thermal cycling induces the degradation of the tooth/restoration interface because of the difference in the thermal expansion coefficient⁽¹³⁾. This methodology is proposed by the International Organization for Standardization in its ISO 11405 standard as a method to accelerate material aging, simulating its survival in the mouth⁽¹⁴⁾.

The simplicity and effectiveness of the enamel adhesion process is well known⁽¹⁵⁾. Of the available adhesive strategies, the three-step etch-and-rinse techniques and the two-step self-etch techniques are considered as the gold standard⁽¹⁶⁻¹⁷⁾. However, the two-step etch-and-rinse technique is the most widely used one in our country, also yielding satisfactory results⁽¹⁸⁻¹⁹⁾.

Objective

To evaluate and compare the relationship between immediate enamel bond strength and the microleakage of two composite resin-based restorative systems through microtensile and microleakage tests.

The hypothesis raised is that the greater the bond strength value, the lower degree of microleakage.

Materials and methods

Obtaining samples

The sample included 40 third molars with a healthy crown regardless of root development status. They were obtained in the surgical block service of the School of Dentistry, having been removed for reasons unrelated to this research. The teeth were collected after the patients signed a written informed consent and upon approval of the project by the Ethics Committee of the School of Dentistry - UDELAR (File no. 215/14). Once extracted, the molars were cleaned with periodontal cures and prophylaxis with pumice stone and water was implemented. They were then stored in 0.5% Chloramine-T for 7 days and subsequently in distilled water at 3°C to 5°C for no more than 3 months. This procedure is protocolized by the Human Tooth Bank of the School of Dentistry (File no. 091100-000933-11, Council of the School of Dentistry).

All restorations were made in the same batch and samples for each trial were randomly distributed (generated through www.randomizer.org) into two groups of twenty molars each, depending on the restorative system used:

- Group "Z": 20 molars restored with Adper Single bond 2+ Filtek Z250 XT (3M SPEC; ST PAUL, MN, USA).
- Group "T": 20 molars restored with Tetric N Bond + Tetric N Ceram (Ivoclar Vivadent; Schaan, Liechtenstein, Germany).

All the materials were handled in compliance with the manufacturer's instructions. Both adhesive systems were applied after conditioning the enamel with 37% phosphoric acid for 15 seconds (Dentsply; Petropolis, RJ, Brazil). The same color (A2) was selected for both restorative systems to avoid having polymerization variables induced by pigments. Both composite resins have nanohybrid filler so this variable was also controlled to avoid biases. Table 1 includes information regarding composition, manufacturer's indications and batch.

Table 1: Batch, composition and handling information provided by the manufacturers of the restorative systems (adhesive + restorative composite resin) used in this study

COMMERCIAL NAME	MANUFACTURER	COMPOSITION	HOW TO USE	BATCH
FILTEK Z250 XT	3M ESPE	Matrix: Bis-GMA, Bis-EMA, TEGDMA, PEGDMA and UDMA Filler: Nanohybrid (silica, zirconia, zirconia/silica cluster). 82% by weight. Silane	The adhesive systems recommended by the manufacturer are: 3M™ Single Bond or 3M™ Scotchbond™ Multi-Purpose Adhesive Systems Incremental application (Increments of up to 2.5 mm) Light-cure for 20 seconds	N793196
TETRIC N CERAM	Ivoclar Vivadent	Matrix: Bis-GMA, UDMA, Bis-EMA, TEGDMA. Filler: Nanohybrid (barium glass, ytterbium trifluoride, silicon dioxide). 63.5% by weight. Silane	The adhesive system recommended by the manufacturer is Tetric N-bond. Incremental application (Increments of up to 2mm) Light-cure for 20 seconds	V16057
ADPER SINGLE BOND 2	3M ESPE	Matrix: Bis-GMA, HEMA, dimethacrylates Filler: Silica nanoparticle (10% by weight) Solvent: ethanol and water	Conditioning with 37% phosphoric acid for 15 seconds Rinse (10 seconds) and dry Application of adhesive Rubbing for 15 seconds Place two layers Drying for 5 seconds Light-cure for 10 seconds	N703454
TETRIC N BOND	Ivoclar Vivadent	Matrix: Bis-GMA, UDMA, hydroxyethyl methacrylate. Filler: Silica nanoparticle (1% by weight) Solvents: ethanol and water	Conditioning with 37% phosphoric acid for 15-30 seconds Rinse (5 seconds) and dry Apply adhesive by rubbing against walls for 10 seconds Drying Light-cure for 10 seconds	U34550

Microtensile test

Half the teeth assigned to each group were used for the microtensile test (n=10). The buccal faces of the molars were worn down with sandpaper with 600 grit size by making eight-shaped movements to achieve a flat, smooth enamel surface. This surface was used to apply the adhesive treatment. It had been conditioned with 37% phosphoric acid for 15 seconds. After that time, the surface was washed vigorously with pressurized water for 15 seconds followed by pressurized air, drying in a direction oblique to the surface. The adhesive system was then applied according to the corresponding group and following the manufacturer's instructions. The adhesive layer was light-cured with an Optilight Max halogen unit (Gnatus; Ribeirao Preto, Brazil) and previously analyzed with a Bluephase Meter radiometer (Ivoclar Vivadent; Schaan, Liechtenstein, Germany) at 980mw/cm².

A 4 mm high and 5 mm wide resin block was then made on the buccal face, incrementally adding 2 mm layers and light-curing each increment according to the manufacturer's instructions. The specimens were stored 24 hours in distilled water at 37°C to allow time for polymerization reactions to end and release the stresses induced by it. Then, using a Gellings-Hamco microtome (Hamco Machines INC.; Rochester, NY, USA) 1 mm² specimens were sectioned (rods), which were half enamel and half composite resin (Fig. 1).

The microtensile test was performed using an MTS SANS CMT 2000 5KN universal testing machine (MTS Systems Corporation; Shanghai China) at a speed of 1mm/min and a 100 N charge cell.

Bond strength (MPa) was calculated using the following formula:

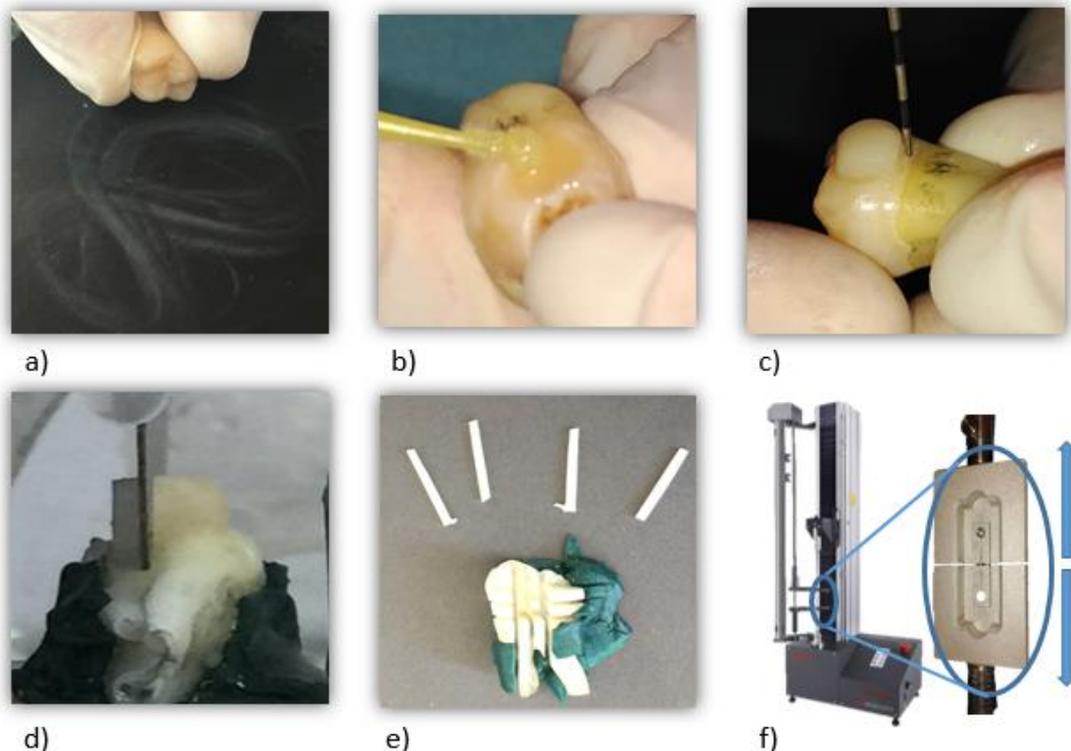
$$\mu TBS (MPa) = F(N)/A(mm^2)$$

Where μTBS is the microtensile bond strength in MPa, F is the maximum force measured by the universal test machine in Newton, and A is the cross-sectional area of the specimen calculated according to width multiplied by depth.

The specimens were subjected to microtensile tests according to the ISO 11405 standard⁽¹⁴⁾. The failure mode was analyzed in each of the samples with a stereoscope (10x) and classified into failures:

- a. Adhesive
- b. Cohesive
- c. Mixed

Fig. 1: Process followed to obtain specimens for microtensile testing



a) Flat and smooth surface on the buccal face. b) Implementing the adhesive strategy. c) Making a composite resin block. d) Sectioning the resin block with the microtome. e) Resulting specimens. f) Microtensile test in universal testing machine.

Microleakage test

A total of 10 teeth per group were used (n=10) for the microleakage test. A cavity was made in enamel and dentin at the middle third of the buccal face using Kavo EXTRATORQUE 505C high-speed rotary instruments with spray (Kavo Dental; Schwaben, Germany) and a 100-110 micron/1.6 mm diameter medium-grain diamond cylindrical bur (Microdont Dental Products; Monsei, NY, USA). We obtained a dentin cavity measuring 3 mm in diameter and 1 mm deep. The bur was changed every three cavities. The cavities were standardized using an American Eagle periodontal probe (American Eagle Instruments Inc.; Missoula, Montana, USA), and if the measurements were not the same, the specimen was discarded. The adhesive treatment of the corresponding group was implemented following the manufacturer's instructions. The composite resin was placed by adding consecutive 2 mm thick layers until the entire cavity was filled. The adhesive and resin were light-cured with a GNATUS Optilight Max unit. Once completed, the restoration was finished with a 30 micron/0.16 diameter extra-fine diamond stone (Microdont Dental Products; Monsei, NY, USA) and polished with medium and extra-fine grain discs (Microdont Dental Products; Monsei, NY, USA) until a smooth surface was obtained. The restored specimens were stored for 24 hours in distilled water at 37°C. Finally, the samples were sealed with transparent Colorama synthetic polish (L'Oréal Brasil; São Paulo, Brazil), applying two layers that covered the root up to the cemento-enamel junction (except 1 mm around the cavo surface angle) in order to avoid possible root or apical filtrations.

For the microleakage test, the samples were subjected to 500 thermal cycles in a thermal cycler (Termocycle; Biopdi, São Paulo, Brazil) at between 5° and 55° C, each bath lasting 20 seconds, with intervals between baths of 10 seconds, following the ISO/TS 11405 standard⁽¹⁴⁾. According to some studies, 500 cycles are sufficient to observe a microleakage effect on the interface, and the increase in the number of cycles is not associated with increased microleakage⁽²⁰⁻²¹⁾. Finally, the samples were immersed in basic fuchsin in 0.5% propylene glycol solution for 24 hours, following previously established protocols⁽²²⁻²³⁾, and sagittally cut with a Gellings-Hamco microtome (Hamco Machines INC.; Rochester, NY, USA) to be evaluated (Fig. 2).

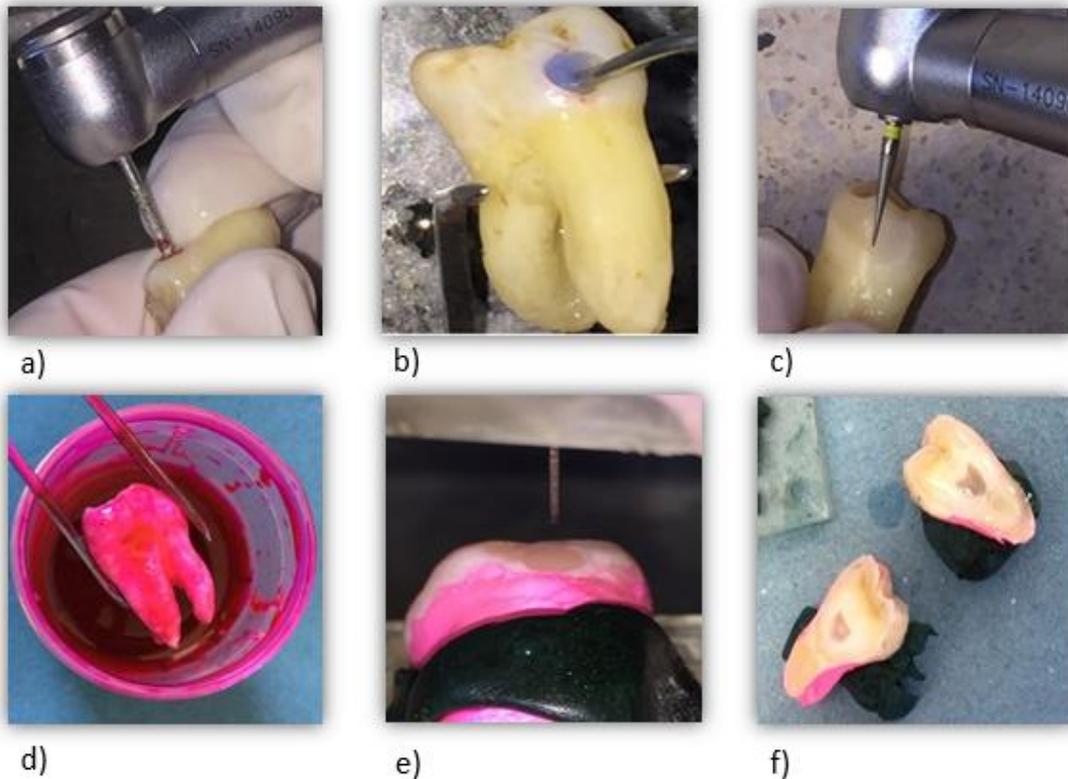
The samples were analyzed with a ROSSBACH YZ-6 stereoscope (Rossbach de Mexico; Mexico City, Mexico), and classified according to the degree of dye penetration:

- 0 - No dye penetration
- 1 - Dye penetrated the enamel walls
- 2 - Dye penetrated the walls, reaching the dentin

3 - Dye reached the floor of the cavity

All the observations were made by the same researcher (See Fig. 3).

Fig. 2: Process followed to obtain specimens for microleakage testing



a) Class 5 cavity drilling on the buccal face. b) Implementation of adhesive strategy. c) Filling with composite resin, finishing and polishing of the surface. d) Immersion in dye after thermal cycling. e) and f) Cutting the samples in half with microtome.

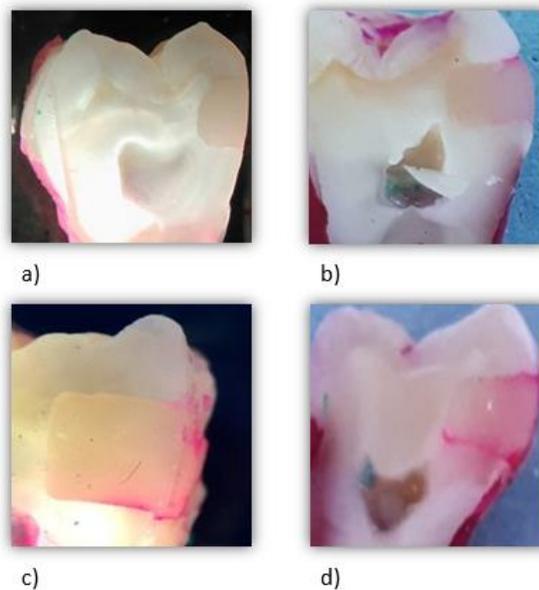
Statistical analysis

Statistical analysis was performed using SigmaPlot Version 12.0. Data obtained about microtensile bond strength were analyzed using Student's t-test. In addition, microleakage values were analyzed using the Mann-Whitney U (non-parametric) test. A Pearson correlation analysis was used to determine the degree of association of the dependent variables studied. A significance level of $\alpha = 0.05$ was chosen for all the analyses.

Results

Microtensile bond strength values are presented in Fig. 4. The results of this study showed a statistically significant difference between the groups ($p = 0.049$); Tetric N Ceram (Ivoclar Vivadent ®) had a higher bond strength value compared to Filtek Z250XT (3M ESPE®). Additionally, no statistically significant differences were found in the degree of microleakage ($P=0.478$) between the two restorative materials (Table 2).

Fig. 3: Degree of microleakage classification



a) Degree 0: no dye penetration. b) Degree 1: dye penetrated the enamel walls. c) Degree 2: dye penetrated the walls, reaching the dentin. d) Degree 3: dye reached the floor of the cavity.

Fig. 4: Mean bond strength values and corresponding standard deviations (SD) obtained after the microtensile test.

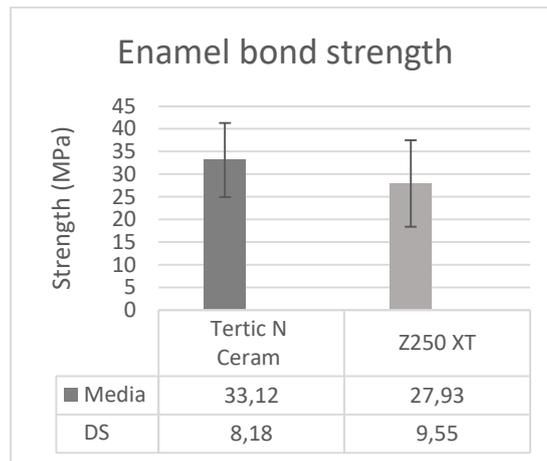


Table 2. Degree of microleakage of the restorative systems evaluated

MICROLEAKAGE	TERTIC N CERAM	Z250 XT
DEGREE 0	5	4
DEGREE 1	2	0
DEGREE 2	1	3
DEGREE 3	2	3
MEDIAN (RI)	0.50 (0.00 - 2.25)	2.00 (0.00 - 3.00)
	p = 0.478	

The correlation analysis showed no statistically significant correlation ($p = 0.196$) between microtensile bond strength and microleakage, and the correlation coefficient was 0.09.

Discussion

Based on the results of this study, it was not possible to accept our hypothesis, since statistically significant differences were found in the bond strength values ($p = 0.049$). This was not the case in the microleakage test, as both resins had a similar degree of microleakage ($p = 0.478$).

We know that adhesive dentistry aims to develop materials that can replace dental structure. Sealing capacity (nanoleakage and microleakage) and adhesive bond strength are two of the main types of laboratory tests used to evaluate the performance of restoration systems⁽²⁴⁾.

Regarding bond strength, the data obtained in this study are consistent with the literature: between 28.57 and 29 MPa for Tetric N Bond^(25,26), and 24.8 to 29.7 MPa for Adper Single Bond 2⁽²⁷⁾.

It is important to mention that this study found no direct relationship between bond strength and microleakage, as have other works in the literature⁽²⁸⁻³¹⁾.

We must also note that the aim of the microleakage test is not to determine material survival, because there is evidence in the literature that penetration tests to evaluate microleakage do not correlate with the clinical success parameters of the restoration (postoperative sensitivity, retention, marginal coloration)⁽³²⁾. The aim of thermal cycling is to simulate the aging of the material while the microleakage test aims to analyze the failure mode of the adhesive interface⁽³³⁾. This is why studies are still conducted aiming to predict the behavior of polymeric restoration systems using this type of test⁽²⁸⁻³¹⁾.

Having discussed the results and after analyzing other scientific studies, we can conclude that bond strength is not the only determining factor when testing the performance of an adhesive system. In addition, multiple other variables inherent to the material must be considered (filler type and percentage, degree of volumetric contraction, hydrolytic stability, thermal expansion coefficient, etc.)⁽³⁴⁻³⁵⁾, the technique (stratified technique, monolayer, C factor, light-curing times and intensity, etc.)⁽³⁵⁻³⁶⁾ the substrate (depth, tissues involved, type of dentition, smear layer removal, etc.)⁽³⁷⁾, and the medium (pH variability, mechanical loads received, etc.)^(36,38-41).

Currently, we can say that restorative systems satisfactorily meet the requirements set out in the standards, achieving good clinical performance. This is why selecting a restorative system has become a minor problem today^(2,9). Additionally, the "operator factor", which is rarely studied, is likely to play a major role throughout the longevity process⁽⁴²⁻⁴⁴⁾.

Therefore, it is essential that professionals know the material and its correct handling (respecting the established protocols) in order to create an effective and long-lasting bond with the tooth structure.

More studies considering these variables must be conducted in order to support our conclusions and consolidate the role of other factors in the performance of adhesive systems, such as long-term clinical trials, which can record real conditions more closely.

Conclusions

The restorative systems used were unable to prevent microleakage, although Tetric N Ceram obtained higher bond strength values.

No connection was found between bond strength values and degree of microleakage. Bond strength is not the only determining factor for predicting the performance of an adhesive system, so other multiple variables inherent to the material, substrate, technique, and environment should be taken into account.

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Conflict of interests

The authors declare that they have no conflict of interest regarding companies mentioned in the study.

Authorship contribution

MELC has contributed in, 1, 2, 3, 4, 5.

MMG has contributed in, 1, 2, 3, 4, 5.

CECS has contributed in, 3, 4, 6.

FMG has contributed in, 1, 4, 6.

GSGP has contributed in, 1, 3, 4, 5, 6.

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