

Influence of Different Surface Treatments on Bond Strength of Resin Composite Using the Intrinsic Characterization Technique

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Clinical Relevance

Surface treatments can adversely reduce the bond strength between resin composite and light-cured characterizing materials. Maintenance of the air-inhibited surface layer of resin composite is still the best alternative for optimizing the bond between resin composite and light-cured characterizing materials, therefore simplifying the clinical steps for performing dental characterization procedures.

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SUMMARY

Objective: This study evaluated the influence of different surface treatments on the resin bond strength/light-cured characterizing materials (LCCMs), using the intrinsic characterization technique. The intrinsic technique is characterized by the use of LCCMs between the increments of resin composite (resin/thin film of LCCM/external layer of resin covering the LCCM).

Materials and Methods: Using a silicone matrix, 240 blocks of composite (Z350/3M ESPE) were fabricated. The surfaces received different surface treatments, totaling four groups (n=60): Group C (control group), no surface treatment was used; Group PA, 37% phosphoric acid for one minute and washing the surface for two minutes; Group RD, roughening with diamond tip; and Group AO, aluminum oxide. Each group was divided into four

subgroups (n=15), according to the LCCMs used: Subgroup WT, White Tetric Color pigment (Ivoclar/Vivadent) LCCM; Subgroup BT, Black Tetric Color pigment (Ivoclar/Vivadent) LCCM; Subgroup WK, White Kolor Plus pigment (Kerr) LCCM; Subgroup BK, Brown Kolor Plus pigment (Kerr) LCCM. All materials were used according to the manufacturer's instructions. After this, block composites were fabricated over the LCCMs. Specimens were sectioned and submitted to microtensile testing to evaluate the bond strength at the interface. Data were submitted to two-way analysis of variance (ANOVA) (surface treatment and LCCMs) and Tukey tests.

Results: ANOVA presented a value of $p < 0.05$. The mean values (\pm SD) for the factor surface treatment were as follows: Group C, 30.05 MPa (± 5.88)a; Group PA, 23.46 MPa (± 5.45)b; Group RD, 21.39 MPa (± 6.36)b; Group AO, 15.05 MPa (± 4.57)c. Groups followed by the same letters do not present significant statistical differences. The control group presented significantly higher bond strength values than the other groups. The group that received surface treatment with aluminum oxide presented significantly lower bond strength values than the other groups.

Conclusion: Surface treatments of composite with phosphoric acid, diamond tip, and aluminum oxide significantly diminished the bond strength between composite and the LCCMs.

INTRODUCTION

Light-cured characterizing materials (LCCMs) are fluid composites containing small load concentrations with colorants or unfilled resins with color tints. They are generally used to characterize occlusal fissures and sulci;^{1,2} cover metal posts, darkened teeth, and teeth with metal pigments;³ and mimic the chromatic characteristics of the tooth.^{1,4} An LCCM may be presented in various pigments, such as brown, black, red, ochre, and white, among others, with the purpose of providing a direct restoration with a more natural and harmonious appearance in relation to the adjacent teeth.⁵

The application of LCCMs may be done by means of two techniques: extrinsic or intrinsic. With the extrinsic technique, the LCCM is applied with an explorer tip or brush after the restoration is finished and polished, with the LCCM flowing into the sculpted sulci. The LCCMs must be applied in thin

films (< 0.2 mm) followed by light activation. However, as LCCMs have low resistance to abrasion because of the smaller (or without) quantity of inorganic load incorporated into the organic matrix, they easily become worn when submitted to occlusal contacts and the restoration soon loses the characterization.⁶⁻⁸

To prevent the early loss of the effect of characterization, it is recommended that LCCMs should be covered with an external layer of resin composite. This modification in the application of LCCMs is what characterizes the intrinsic technique. With the intrinsic technique, LCCMs are used between the increments of resin composite. The LCCMs are applied and light activated on the previously light-activated resin composite; they are then covered with one or more layers of resin composite until the restoration is finished.⁹ The average thickness of the additional layer(s) varies between 0.2 and 0.5 mm. Pucci and colleagues¹⁰ observed that the intrinsic technique may lead to adverse alteration in the physical or mechanical properties of resin composite, weakening the resin composite/LCCMs/resin composite interfaces, which may promote early failure and compromise the durability of the restoration.

Traditionally, resin composites are compounds with dimethacrylate monomers that cure via a free-radical-induced polymerization. This free-radical polymerization reaction can be inhibited by atmospheric oxygen, resulting in a superficial layer of soft, sticky, liquid-like consistency and poorly polymerized monomer, referred to as an oxygen-inhibited layer. The oxygen inhibited layer is beneficial to interfacial resin-to-resin bonding during incremental technique because interfacial bonding can be increased due to the consistency of the oxygen-inhibited layer which increases the contact area between two contacting polymer increments.^{11,12} Additionally, the oxygen-inhibited layer can let the polymers of both layers combine to form an interdiffused zone. This zone is marked by the formation of chemical bonds due to copolymerization.^{11,12}

To increase the bond strength between the layers of resin, weakened by the use of the LCCMs, a possible alternative would be to perform surface treatment on the resin composite to optimize the resin composite/LCCM bond. Therefore, the aim of this study was to evaluate the influence of different surface treatments on the bond strength of resin composite and LCCMs using the intrinsic characterization technique.

Table 1: *Materials Used and Their Compositions*

Material	Manufacturer	Composition
Diamond tip 4103	KG Sorensen, Cotia, SP, Brazil	Stainless steel and grains of natural diamond with controlled dimensions.
Magic Acid Gel	Vigodent, Rio de Janeiro, RJ, Brazil	37% phosphoric acid
Airborne particle abrasion with aluminum oxide particles	Microetcher ERC, Danville Engineering, San Ramon, CA, USA	Aluminum oxide of 50 μm
Resin composite Filtek Z350	3M ESPE, St Paul, MN, USA	Bis-GMA (bisphenol A-glycidyl-methacrylate), UDMA (urethane dimethacrylate), TEGDMA (triethyleneglycol dimethacrylate), Bis-EMA (glycidyl ethoxylate dimethacrylate), 20 nm nanosilica filler, agglomerates of primary zirconia/silica particles with 5-20 nm fillers (78.5% by weight)
Kolor Plus	Kerr, Orange, CA, USA	Uncured methacrylate ester monomers, inert mineral fillers, photoinitiators, and stabilizing additives
Tetric Colors	Ivoclar/Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, and TEGDMA (86% by weight); silanized silicone dioxide (12%–13 % by weight); catalyzers, stabilizers, and pigments (<2% by weight)

METHODS AND MATERIALS

Two hundred and forty blocks of resin composite (Filtek Z350/shade A3, 3M ESPE, St Paul, MN, USA) measuring $4 \times 4 \times 4$ mm were fabricated with the use of a silicone matrix. The composite, shade A3, was inserted into the silicone matrix using the incremental technique and light polymerized for 40 seconds per increment at an intensity of 500 mW/cm^2 (Curing Light XL 3000, 3M Dental Products, St Paul, MN, USA).

The resin composite blocks were divided into four groups ($n=60$) according to the type of surface treatment performed:

Group 1: control group, no surface treatment was used;

Group 2: Composite surface was treated with 10% phosphoric acid (Magic Acid Gel, Vigodent, Rio de Janeiro, RJ, Brazil) for one minute, followed by water rinsing for two minutes and air-drying;

Group 3: Composite surface was treated with diamond tip 4103 (Kg Sorensen, Cotia, SP, Brazil) standardized by five repetitions;

Group 4: Composite surface was sandblasted with $50 \mu\text{m}$ aluminum oxide particles (Micro-etcher ERC,

Danville Engineering, San Ramon, CA, USA) for 10 seconds.

Next, each group was divided into four subgroups ($n=15$), according to the type of LCCM used:

Subgroup WT: Application of White Tetric Color pigment LCCM (Ivoclar/Vivadent, Schaan, Liechtenstein);

Subgroup BK: Application of Brown Kolor Plus pigment LCCM (Kerr, Orange, CA, USA);

Subgroup WK: Application of White Kolor Plus pigment LCCM (Kerr);

Subgroup BT: Application of Black Tetric Color pigment LCCM (Ivoclar/Vivadent).

For application of the surface coloring agent, a silicone mold measuring 4×4 mm and 0.4 mm high was used. The coloring agents were applied with a microbrush and light polymerized for 40 seconds at an intensity of 500 mW/cm^2 (Curing Light XL 3000).⁶ The light unit tip was at a distance of 5 mm from the coloring agents. The materials used in the study and their compositions are listed in Table 1.

On the light-activated coloring agents, resin composite blocks (Filtek Z350/shade A3, 3M ESPE)

Table 2: *Results of ANOVA for Three Factors*

Factor	Degrees of Freedom	F	p
Surface treatment	3	18.47	0.000*
light-cured characterizing material (LCCM)	3	2.51	0.0656
Surface treatment \times LCCM	9	4.37	0.0001*

* Significant differences.

measuring $4 \times 4 \times 4$ mm were made with the use of a silicone matrix. The specimens were immersed in water at 37°C for 48 hours.

After this, the specimens were submitted to thermomechanical wear (ER 37000, Erios, São Paulo, SP, Brazil). Mechanical cycling was performed with a 60N load and 100,000 cycles, with the force applied on the specimen perpendicular to the surface at the resin/LCCM interface. Simultaneously, the specimens were submitted to 100,000 cycles of thermal cycling at temperatures of 5°C, 37°C, and 55°C for 30 seconds each.

Parallel sections measuring approximately 1 mm were made using a diamond disc attached to a Labcut 1010 (Extec Technologies Inc, Perris, CA, USA) cutting machine. Sections were made at low speed under water cooling to prevent stress induction at the bond interface.

Specimens (≈ 6 sticks per block) were attached to a microtensile device in a universal testing machine EMIC (DL-1000, São José dos Pinhais, PR, Brazil) with a 10 kg load cell at a crosshead speed of 1 mm/

min, according to the ISO 11405 Standard (Dental materials – Guidance on testing of adhesion to tooth structure). Data, expressed in megapascals (MPa), were submitted to parametric two-way analysis of variance (ANOVA) (surface treatment and LCCM) and Tukey post-hoc test, at a 5% level of significance.

The fractured specimens were analyzed under a stereomicroscope Stemi 2000 (Karl Zeiss, Jena, Germany) at 50 \times magnification for determining fracture type: adhesive, fractures in which the failure occurred at the composite (first block)/surface treatment/LCCM interface in more than 75% of the analyzed area; cohesive in resin, fractures in which the failure occurred at the LCCM/composite interface (second block, without surface treatment) or only in resin composite in more than 75% of the analyzed area; or mixed, fractures for which there was no predominance greater than 75% of any type of failure.

RESULTS

The results of three-way ANOVA are shown in Table 2. ANOVA showed a value of $p < 0.05$ for the surface treatment factor and interaction between factors, which indicated that there were significant differences among the groups.

The bond strength means (\pm SD) and the results of Tukey test for the factor surface treatment are shown in Table 3. The control group presented significantly higher bond strength values than the other groups. The groups in which surface treatment was performed with the diamond tip and acid etching presented significantly higher bond strength than the group in which aluminum oxide was used.

The bond strength means (\pm SD) and Tukey test results for the interaction between factors are shown in Table 4. The control group associated with the White Kolor Plus pigment and Black Tetric Color pigment LCCMs presented significantly higher bond strength values than the group in which surface

Table 3: *Mean Bond Strength Values (\pm SD) for the Factor Surface Treatment and Tukey Test Results for All Groups*

Surface Treatment	Mean MPa Values (\pm SD)	Homogeneous Groups*
Control	30.05 (\pm 5.88)	A
Phosphoric acid	23.46 (\pm 5.45)	B
Diamond tip	21.39 (\pm 6.36)	B
Aluminum oxide	15.05 (\pm 4.57)	D

* Means accompanied by the same letters presented no statistically significant differences.

Table 4: Mean Bond Strength Values (\pm SD) for the Interaction Between the Factors and Tukey Test Results for All Groups/Subgroups

Surface Treatment	Light-cured Characterizing Material	Mean MPa Values (\pm SD)	Homogeneous Groups*			
Control	Kolor Plus White	34.45 (\pm 5.88)	A			
Control	Tetric Color Black	33.75 (\pm 4.06)	A			
Control	Tetric Color White	28.93 (\pm 4.14)	A	B		
Phosphoric acid	Kolor Plus Brown	27.81 (\pm 5.13)	A	B	C	
Diamond tip	Tetric Color Black	25.38 (\pm 5.62)	A	B	C	D
Phosphoric acid	Tetric Color White	24.62 (\pm 3.58)	A	B	C	D
Diamond tip	Tetric Color White	24.19 (\pm 5.87)	A	B	C	D
Aluminum oxide	Kolor Plus White	23.26 (\pm 5.51)	A	B	C	D
Control	Kolor Plus Brown	22.34 (\pm 6.08)	A	B	C	D
Phosphoric acid	Tetric Color Black	21.35 (\pm 6.01)		B	C	D
Diamond tip	Kolor Plus White	20.75 (\pm 5.48)		B	C	D E
Phosphoric acid	Kolor Plus White	19.67 (\pm 3.55)		B	C	D E
Aluminum oxide	Tetric Color White	17.76 (\pm 4.90)		B	C	D E
Diamond tip	Kolor Plus Brown	15.72 (\pm 5.0)			C	D E
Aluminum oxide	Kolor Plus Brown	13.89 (\pm 3.7)				D E
Aluminum oxide	Tetric Color Black	8.75 (\pm 2.74)				E

* Means accompanied by the same letters presented no statistically significant differences.

treatment was performed with phosphoric acid associated with the Black Tetric Kolor and White Kolor Plus LCCMs, than the group in which surface treatment was performed with a diamond tip associated with the White Kolor Plus and Black Kolor Plus LCCMs, and than the group in which surface treatment was performed with aluminum oxide associated with the White Tetric Kolor, Brown Kolor Plus and Black Tetric Color LCCMs. The control group associated with the White Tetric Color LCCM presented significantly higher bond strength values than the group in which surface treatment

was performed with the diamond tip associated with the Brown Kolor Plus LCCM and than the groups in which surface treatment was performed with aluminum oxide associated with the Brown Kolor Plus and Black Tetric Color LCCMs.

Figure 1 shows the representative graph of the mean bond strength values for all the groups/subgroups.

With regard to the fracture results, the values shown in Table 5 were obtained. Adhesive fractures at the resin/surface treatment/LCCM interface were higher than the cohesive fractures (LCCM/composite

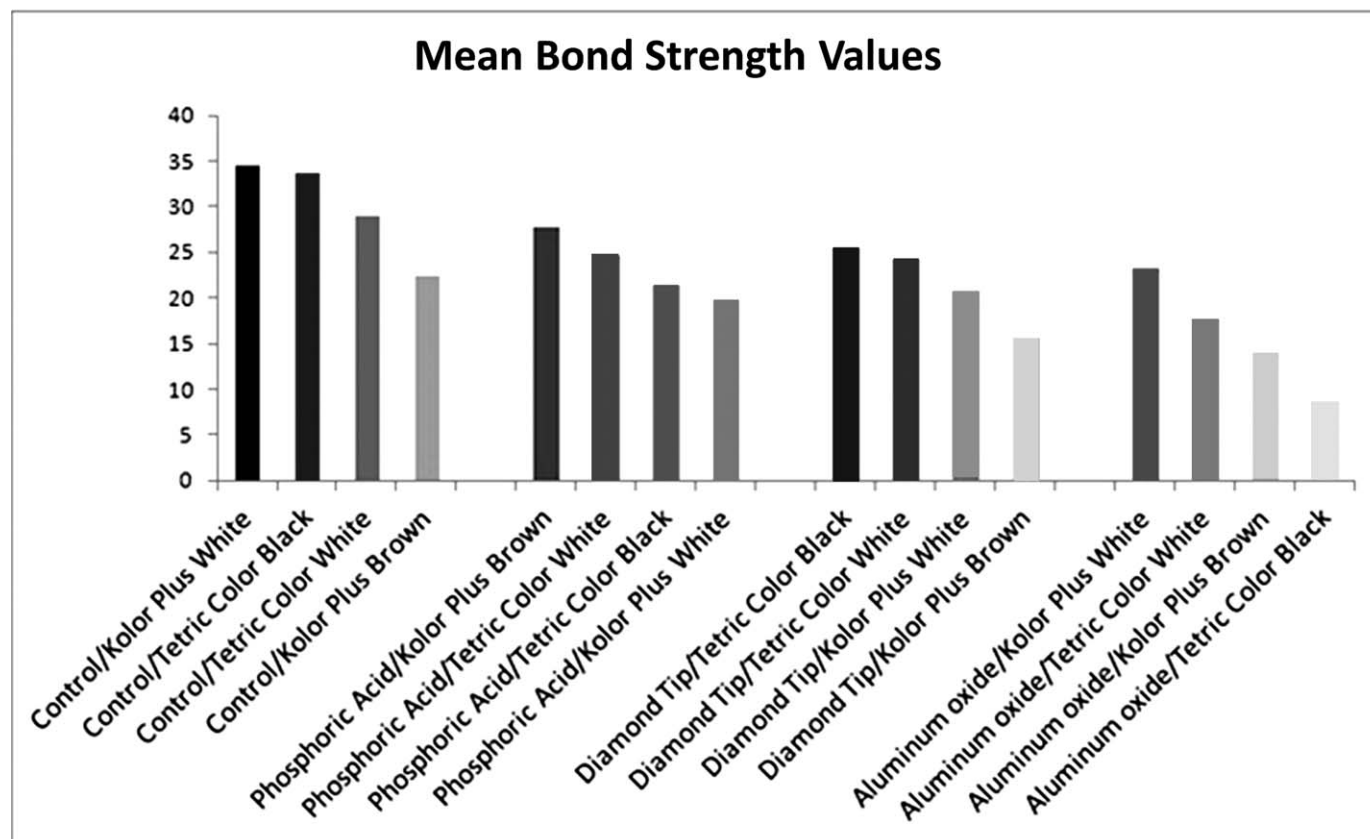


Figure 1. Representative graph of the mean bond strength values for all the groups and subgroups.

or only in composite) and suggest failure in the adhesive process.

DISCUSSION

Different surface treatments have been reported for improving the bond between the repair resin composite and the existing resin composite restoration. Roughening composite bonding surfaces with high-speed diamond burs can produce irregularities on the resin composite surface. This has resulted in an increased surface area for micromechanical retention, thereby increasing the repair bond strength.¹³ Sandblasting with aluminum oxide increases the surface irregularity and wetting potential of the repair composite,¹³ and sandblasting with aluminum oxide is an efficient and cost-effective procedure for repair of aged composite restorations.¹⁴ Surface treatment with phosphoric acid at 37% removes residues on the surface, which facilitates the bond between layers of composites.^{15,16} It also seems to remove the organic contamination of the composite surface.¹⁷

The intrinsic characterization technique of resin composites using surface LCCMs is routinely used in

the dental office. In 2011, Pucci and colleagues¹⁰ observed that the use of Tetric Kolor (White and Black pigments) and Kolor Plus (White and Brown pigments) LCCMs with the intrinsic technique significantly reduced the bond strength between the resin composite layers. Therefore, the aim of this study was to observe whether different resin composite surface treatments used to improve repair procedures could optimize the bond strength between resin composite and LCCMs used in the intrinsic technique.

The results of the present study demonstrated that the control group, in which no surface treatment was performed on resin composite before the use of LCCMs, presented higher bond strength values compared with the groups in which surface treatment was performed with phosphoric acid, diamond tips, and aluminum oxide. LCCMs are resin composites that have a high concentration of organic matrix and small concentrations of load. Resin composite polymerization begins with chemical chain reactions by breaking apart the double carbon bonds for the formation of polymers.¹⁸ The bond strength between successive increments of

Table 5: Classification With Regard to Fracture Type (Number of Sticks)

Surface Treatment	Light-cured Characterizing Material	Adhesive Fracture	Cohesive Fracture	Mixed	Sticks Lost
Control	Tetric Kolor White	33	36	27	0
Control	Tetric Kolor black	30	39	27	3
Control	Kolor Plus White	33	36	30	0
Control	Kolor Plus Brown	45	39	24	0
Phosphoric acid	Tetric Kolor White	99	0	0	0
Phosphoric acid	Tetric Kolor black	63	36	0	3
Phosphoric acid	Kolor Plus White	57	36	3	0
Phosphoric acid	Kolor Plus Brown	75	27	0	0
Diamond tip	Tetric Kolor White	66	24	0	0
Diamond tip	Tetric Kolor black	90	0	0	3
Diamond tip	Kolor Plus White	63	24	3	0
Diamond tip	Kolor Plus Brown	60	39	0	0
Aluminum oxide	Tetric Kolor White	57	30	0	0
Aluminum oxide	Tetric Kolor black	63	27	0	6
Aluminum oxide	Kolor Plus White	57	33	3	0
Aluminum oxide	Kolor Plus Brown	54	30	0	3
Total		945	456	117	18

resin composite occurs because of the presence of an oxygen-inhibited layer of polymerization. This layer is viscous and has unreacted methacrylates that, by means of covalent bonds, will link to the polymer chains, optimizing the bond strength between substrates.^{19–21}

The groups in which surface treatment was performed with phosphoric acid, diamond tips, and aluminum oxide presented lower bond-strength values than the control group because of the removal of the oxygen-inhibited layer, leaving the inorganic matrix on the surface without unreacted methacry-

lates^{19–21} and negatively influencing the bond strength between the substrates. Therefore, these techniques are proposed for improving the bond to the repair of aged resin composite and fresh resin composite.

The results of the present study demonstrated that the group in which surface treatment was performed with aluminum oxide presented significantly lower bond strength values than the groups in which surface treatment was performed with the diamond tip or phosphoric acid. The purpose of surface treatment by the mechanical method (air-

borne aluminum oxide particle abrasion and diamond tip) is to create porosities on the surface to increase micromechanical retention between the layers of the substrate. The objective of chemical surface treatment using phosphoric acid is to perform cleaning of the resin surface to be repaired, thereby improving the bond strength between the resin composite layers.

Some authors^{22,23} have affirmed that airborne aluminum oxide particle abrasion promotes greater irregularity on the surface of the substrate to be repaired, when compared with the use of a diamond tip alone. Consequently, airborne particle abrasion increases the surface area, optimizing micromechanical retention between the resin composite layers.^{22,23} In addition, several authors have affirmed that the use of airborne aluminum oxide particle abrasion could result in a bond strength close to the cohesive strength of the original resin.^{22,23}

However, the oxygen inhibited layer of polymerization is viscous. The reduction in bond strength obtained with surface treatment with aluminum oxide could probably be attributed to the adherence of aluminum oxide particles on this surface layer with unreacted methacrylates, which prevented an effective bond between the LCCMs and resin composite.

The results of the present study also demonstrated that the control group associated with Kolor Plus White and Tetric Color Black LCCMs presented significantly higher bond strength values than the groups in which surface treatment was performed with phosphoric acid associated with Tetric Kolor Black and Kolor Plus White LCCMs, than the groups in which surface treatment was performed with a diamond tip associated with Kolor Plus White and Kolor Plus Black LCCMs, and than the groups in which surface treatment was performed with aluminum oxide associated with Tetric Kolor White, Kolor Plus Brown, and Tetric Color Black LCCMs. As previously described, this may be because removal of the surface layer of inhibited air induced greater fragility of this layer, which negatively influenced the bond strength between the substrates.¹⁸⁻²¹

In addition, the control group associated with the Tetric Color White LCCM presented significantly higher bond strength values than the group in which surface treatment was performed with the diamond tip associated with the Kolor Plus Brown LCCM and than the groups in which surface treatment was performed with aluminum oxide associated with the

Kolor Plus Brown and Tetric Color Black LCCMs. Therefore, these results indicate the importance of the presence of an oxygen-inhibited layer of polymerization to improve the bond strength between successive increments of resin composite.¹⁸⁻²¹

Furthermore, our results showed that the control group associated with the Kolor Plus Brown LCCM was grouped differently from the other control groups. This result might be attributed to the color of the pigment present in the composition of the Kolor Plus Brown LCCM; according to Pucci and colleagues,¹⁰ the darker-pigmented LCCMs showed a greater influence between layers of composites decreasing the cohesive strength of the composite. Beyond that, even though the Kolor Plus Brown LCCM has an organic matrix similar to the composites, the quantity of inorganic filler or the quantity of the pigment present in this material may have affected the cohesive strength of the Kolor Plus Brown LCCM and composite more than the others LCCMs tested. Also, there may be some incompatibility between darker pigment and organic filler in these LCCMs, which could reduce radical polymerization of methacrylate C=C bonds. Unfortunately, the manufactures do not provide these specifications of the LCCMs studied.

With regard to the fracture type, it was observed that the adhesive fractures at the resin/surface treatment/LCCM interface were predominant to the cohesive fractures (LCCM/composite or only in composite) in the groups in which surface treatment was performed. Therefore, these results suggest that there was failure in the adhesive process at the interface in which surface treatment occurred, probably because of the removal of the surface layer of inhibited air, inducing greater fragility of this layer.¹⁸⁻²¹

In addition, it could be observed that the cohesive fracture modes were more evident with the control groups than the surface treatment groups. These results confirm the statistically higher cohesive force among the resin interfaces for control groups without surface treatment, probably because of the presence of an oxygen-inhibited layer of polymerization, which forms a better bond at the interfaces.¹⁸⁻²¹

The results of the present study demonstrated that surface treatments with diamond tips, aluminum oxide, and phosphoric acid significantly reduced the bond strength between the resin composite and LCCMs. Maintenance of the air-inhibited surface layer of resin composite is still the best alternative for optimizing the bond between resin

composite and LCCMs, therefore simplifying the clinical steps for performing dental characterization procedures.

CONCLUSION

According to the methodology used and the data obtained, it may be concluded that surface treatments of composite with phosphoric acid, diamond tips, and aluminum oxide significantly diminished the bond strength between resin composites and LCCMs. The groups in which surface treatment was performed with the diamond tip and acid etching presented significantly higher bond strength than the group in which aluminum oxide was used.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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