ORIGINAL ARTICLE

INFLUENCE OF DIFFERENT SURFACE TREATMENTS ON THE BOND STRENGTH BETWEEN COMPOSITES AND RESIN CEMENT

INFLUÊNCIA DE DIFERENTES TRATAMENTOS DE SUPERFÍCIE NA RESISTÊNCIA DE UNIÃO ENTRE COMPÓSITOS E CIMENTO RESINOSO

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Resumo

O objetivo deste estudo foi avaliar a influência de tratamentos de superfície na resistência de união (RU) entre compósitos e um cimento resinoso. Setenta e cinco discos (10x2 mm) das resinas Filtek P90, Filtek Z250 e Filtek Z350 (3M ESPE) foram divididos em 5 grupos de acordo com o tratamento: N= sem tratamento; S= jateamento com óxido de alumínio (50µm); SE= jateamento de óxido de alumínio + 99,3% de etanol por 5 min; C= jateamento de sílica com Cojet - 30 microns (3M ESPE); CS= jateamento de sílica + silano. Tubos de PVC (0,5 x 0,80 mm) foram fixados nos discos e o cimento resinoso (RelyX ARC, 3M ESPE) foi inserido. Após 24 horas de armazenamento em saliva artificial a 37oC, os espécimes foram submetidos ao teste de microcisalhamento com velocidade de 1,0 mm/min. Os dados foram avaliados em ANOVA de dois fatores e no teste de Tukey (5%) para contraste. Os resultados mostraram que o tratamento com oxido de alumínio (J) foi eficiente no aumento da RU nos compósitos Filtek Z350 e P90. Não houve diferença entre tratamentos para a Z250. Grupo CS mostrou resultados semelhantes aos do controle para todos os compósitos. Já o SE mostrou os piores resultados de RU. Concluiu-se que os valores de RU foram dependentes do tipo de compósito e do tratamento de superfície utilizado. O jateamento com óxido de alumínio parece ser um tratamento de superfície eficaz e pode elevar os valores de RU, já o uso de etanol pode ser prejudicial.

Palavras-chave: Cimentos de resina. Bis-Fenol A-Glicidil Metacrilato. Resinas de silorano. Materiais dentários. Adesivos dentinários.

Abstract

The aim of this study was to evaluate the influence of different surface treatments on bond strength (BS) between composite and a resin cement trough microshear bond strength test. Seventy five discs (10x2 mm) of Filtek P90, Filtek Z250 and Filtek Z350 XT (3M ESPE), were divided into 5 groups according to the treatment: C= control - no treatment; sandblasting J= aluminum oxide (50µm); sandblasting JE = + 99.3% ethanol for 5 min; silica coating S = (3M-ESPE Cojet - 30 microns); SS = silica coating + silane. PVC tubes (0.5 x 0.80 mm) were attached on the composite disc, and then, inserted resin cement (3M ESPE-RelyX ARC). After 24 hours artificial saliva storage at 37oC, the specimens were tested for microshear crosshead speed of 1.0 mm/min. Data were evaluated in two-way ANOVA and Tukey's test (5%) for contrast. The results showed that sandblasting with aluminum oxide (J) was efficient in increasing the BS for composites Z350 and P90. For the Z250, there were no difference between treatments. Also, CS showed results similar to controls for all composites. SE showed the worst results for Z350 e P90. BS values were dependent on the type of composite and the surface treatment used. Sandblasting with aluminum oxide seems to be an effective surface treatment for composites and may lead to higher BS values, while the use of ethanol could be harmful.

Keywords: *Resin cement. Composite resin. Bisphenol A-Glycidyl Methacrylate. Silorane resins. Dental materials. Dentin-Bonding agents.*

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INTRODUCTION

The advancement of adhesive systems allowed composites to be used as direct and indirect restorations. Aesthetics requirements and simpler clinical protocols and techniques associated with satisfactory mechanical strength, extended their use to filling core with and/or without prefabricated post. In these cases, commonly, composites like microhybrids and nanofillers are the most chosen due their better mechanical strength.

Regardless which resin composite is chosen as a filling material, a great bond within this composite and the resin cement, and between the resin cement and the ceramic material of the indirect restoration is needed, providing better retention, marginal sealing and longevity of the restoration. However, the exposition of the filling material to saliva and temporary cements of the interim restorations might affect this bond. Moreover, the non-polymerized monomers from the uppermost surface layer could bind to atmospheric oxygen, reducing the number of binding sites for the resin cement (1, 2).

In the attempt to improve this bond, many chemical and mechanical surface treatments have been tested with the purpose of increasing the surface bond strength of the composite (1, 3-7). Among mechanical treatments, aluminium oxide sandblasting (6-12) and the silica-modified aluminium oxide particles sandblasting (5-7, 10, 12, 13) have shown the most effective results in raising the values of bond strength between composites. Many times, those values reach similar tensile strength values of the original composite (11, 14). However, some studies show an ineffectiveness of these procedures, indicating the need for further researches and investigation of more effective surface treatments (15, 16).

With respect to chemical treatments, it has been stated that ethanol, a solvent of organic matrices, might soften the composite due to its solubility parameter, which is close to the methacrylic polymers ones (17, 18). It was also speculated, in previous studies, that the solvent present in dental adhesives could soften and gel surface and sub-surface composites (19), making it easier for the adhesive system monomers to penetrate the matrix, allowing the establishment of connections within the resin matrix and the filler particles of the composite.

The filling material composition can also affect the bond strength, since each composite class could respond differently after surface treatments (20). Studies have evaluated the repair bond strength in silorane based composites after surface treatments (11-13, 21-23) and the repair bond strength between resin composites (6, 7, 10-12, 22-26). However, few studies approached surface treatments between composites and resin cements by luting analyses with composites blocks for indirect restorations (16, 27) and not by direct analysis of bond strength between a resin cement and a resin composite (9).

Literature about the bond strength between resin composite and resin cement and the possibility of the use of ethanol as a surface treatment agent is scarce. Therefore, the aim of the present study was to determine the influence of surface treatments on the bond strength between different composites and a resin cement. The hypothesis tested was that different surface treatments would affect the bond strength between resin composites and a resin cement.

METHODS

Seventy five disk-shaped resin composites specimens were made using a split metal mold (10 mm diameter x 2mm height), divided into 50 methacrylate-based composite disks (Filtek Z350 XT e Z250) and 25 silorane-based composites (Filtek P90). After cleaning, the resin composite was inserted with a Suprafill spreader (Duflex, RJ, Brazil), filling the entire mold. A polyester strip and a glass slide were positioned above the matrix-composite set and a 65g metal weight was applied for 30s, in order to plan and drain the composite excess. The specimens were photopolymerized (450mW/cm2) for 40s using a halogen photopolymerization unit (Optilux 501, Demetron Kerr, CA, USA). After removing from the mold, the composites disks were storage in artificial saliva inside a dark plastic recipient. After storage time, the disks were embedded in epoxy resin with the bonding surface exposed. All specimens were wet ground down to 150, 300 and 600 silicon carbide paper (DPU-10 Struers, Copenhagem, Denmark). The specimens were cleaned with destilled water, dried and randomly divided to one of the surface treatments protocol (N=75; n=5 per group), as shown in Table 1.

Table 1 - SURFACE TREATMENTS

Group	Treatment
N	No treatment, without surface treatment
S	sandblasted with 50µm aluminium oxide for 20s, 15mm
SE	sandblasted with 50 µm aluminium oxide 20s + absolute ethanol for 5 min
C	30 µm Silica Coating for 20s, 15mm
CS	30 µm Silica coating 20s, 15mm + silane

In the control group (N), no surface treatment was performed. For S group, specimens were airbone particle abraded using a intraoral air-abrasion device (Microetcher, Danville Engineering, USA), sandblasted with 50µm aluminium oxide perpendicular to the surface for 20s from an approximately distance of 15 mm in linear motions at 2.8 bar. After air abrasion, the surface was cleaned with destilled water and dried with air spray. For SE group, the especimens were sandblasted with 50 µm aluminium oxide, as described before. After sandblasting, a cotton pellet soaked in ethanol (absolute ethyl alcohol, 99.3o INPM) was applied for 5 min, dripping a drop each 30s. Groups C and CS were air abraded with 30 µm Silica Coating (Cojet Sand, 3M ESPE, St Paul, MN, USA) for 20s, within 15mm distance, approximately, and 2.8 bar. In the CS group, the silica coting abrasion was followed by the application of two layer of silane (Silane Primer + Activator- Dentsply, Petrópolis, Brazil).

All methacrylate-based composites were conditioned with phosphoric acid for 15s. The surfaces were washed with distilled water for 30s and air dried. After acid conditioning, two layers of the adhesive system Adper Single Bond 2 (3M ESPE, St Paul, MN, USA) were applied. A gentle air blow was applied and the surface was photoactivated for 20s. For Filtek P90, the primer was applied for 15s, gently air dried, and photoactivated for 10s, followed by the bond application and 10s of light curing. PVC tubes (0,5mm height × 0,8mm internal diameter) were obtained from a tracheal suction catheter number 4 (Medsonda, Araponga, PR, Brazil), serially sectioned through a guillotine. A double-sided tape was positioned on the disks surface and 5 PVC tubes were fixed on that tape. RelyX Arc resin cement (3M ESPE, St Paul, MN, USA) was handled and inserted inside the PVC tubes with an elongation tip (Elongation tip, 3M ESPE, St Paul, MN, USA) engaged in a centrix tip (Centrix Accudose Posterior HV, Nova DFL, Rio de Janeiro, Brazil). After removing the excess, the resin cement was photoactivated for 40s. The specimens were storage in artificial saliva inside a dark pot for 24h at 37o C (Figure 1).

Specimens were mounted in a metal jig of the universal testing machine (Emic DL 2000, EMIC Equipamentos e Sistemas de Ensaio; São José dos Pinhais, PR, Brazil), with a 50 N load cell. A chisel (5,22mm width × 0,6mm thickness) applied a shear force to the bond interface at a crosshead speed of 1,0 mm/min until failure. Failures modes were identified using a stereomicroscope at 40x magnification (FZ40, Olympus), and divided as follows: I- adhesive failure of the cement/composite; II- cohesive failure of the composite; III- cohesive failure of the cement; IV- mixed failure. The software Statgraphics 5.1 was used for statistical analyses. As the data was normally distributed and homogeneity, the bond strength values were submitted to two-way ANOVA and Tukey's test at 5% level of significance.

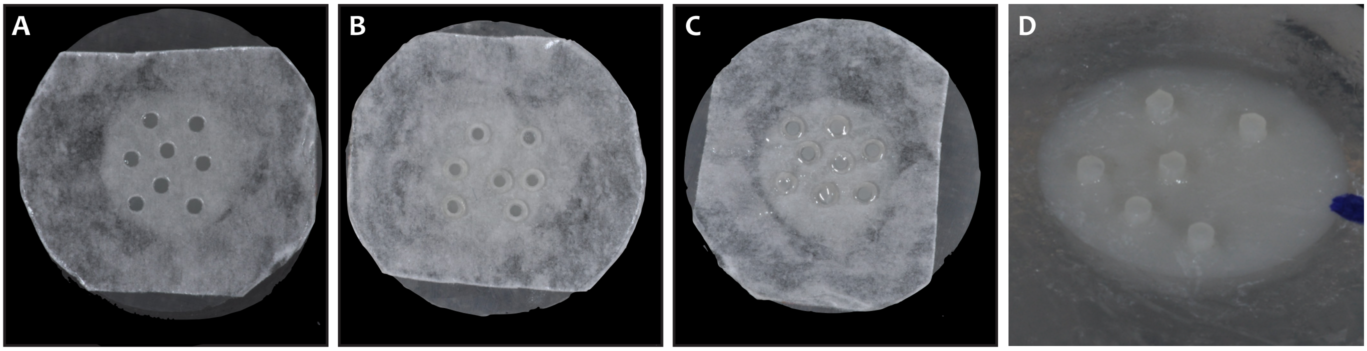

Figure 1 - (A) double-sided tape over the resin composite disk; (B) PVC tubes positioned over the surface; (C) PVC tubes filled with resin cement; (D) After storage and tubes removal, obtaining the specimens.

RESULTS

Analysis of the results demonstrated that individual factors (composite and surface treatment) ($p<0.001$) and the interaction between them ($p<0.01$) were statistically significant. The Tukey's test (5%) could evaluate the differences as are present below (Figure 2)

For P90 composite the oxide aluminum sandblasting was the most effective surface treatment. Ethanol application after sandblasting reduced the bond strength values, showing values similar to control group. The silica sandblasting with silane application was similar to oxide sandblasting, however similar to control group. The values of silica sandblasting without silane, reduced the bond strength becoming similar to control group.

For Z250 composite, no treatment was effective, because all of them were similar to control (N=S=C=CS) or lower bond strength values (SE<C). And nanofiller composite Z350, the oxide aluminum sandblasting was the most effective treatment, while silica coating alone or with silane application were near to control group. For this composite, the ethanol after sandblasting reduced to the lowest bond strength values.

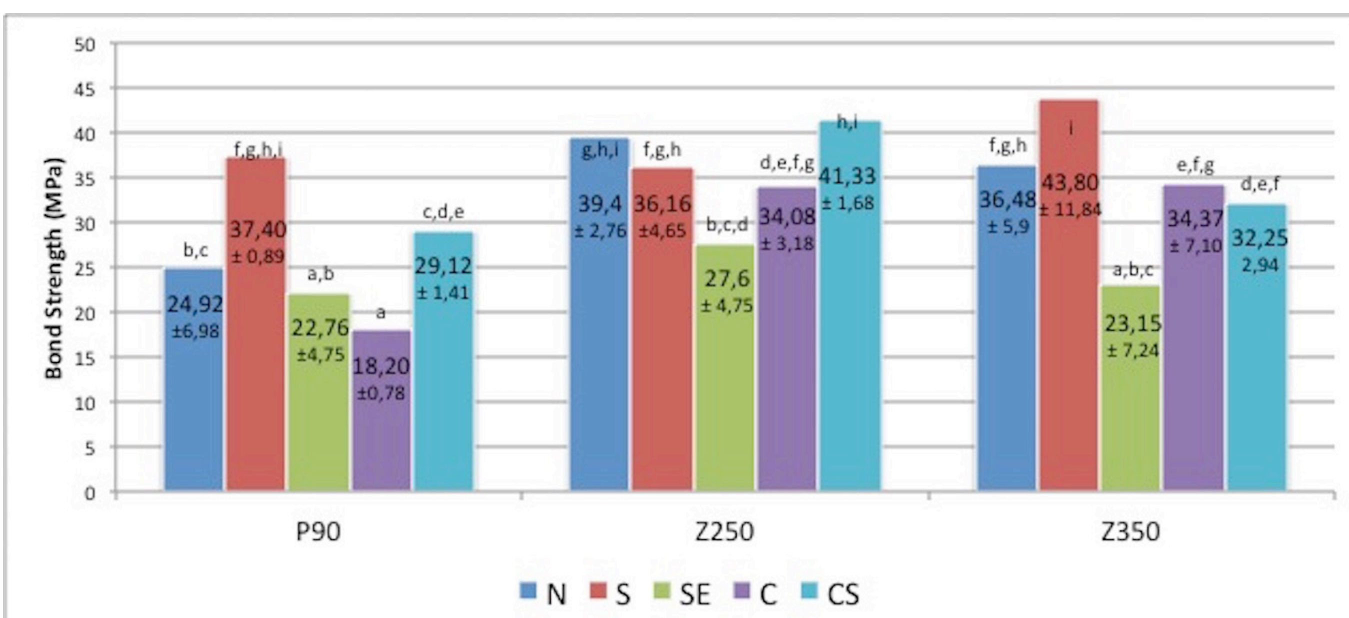

Figure 2 - Means and standard deviations of the bond strength (MPa) between composites and resin cement

Analyzing the failure pattern, the experimental groups with the lowest bond strength as P90-C, P90-SE, Z250- SE and Z350-SE showed more adhesive failures and cohesive of resin cement. The groups with higher bond strength values, showed more cohesive failure of resin composite.

Table 2 - RESULTS OF THE PATTERN OF FAILURE ANALYSES

Group	ADHES (I)	COHES-com (II)	COHES-cem (III)	MIX (IV)
P90 N	17	0	8	0
P90 S	4	14	3	4
P90 SE	16	9	0	0
P90C	16	7	0	2
P90 CS	3	16	0	6
Z250 N	4	15	2	4
Z250 S	4	14	2	4
Z250 SE	18	0	5	2
Z250 C	2	15	4	4
Z250 CS	2	21	0	2
Z350 N	5	13	0	7
Z350 S	0	21	0	4
Z350 SE	18	0	3	4
Z350 C	0	17	2	6
Z350 CS	5	16	4	0

I- adhesive failure of the cement/composite; II- cohesive failure of the composite; III- cohesive failure of the cement; IV- mixed failure

DISCUSSION

In the present study, the microshear test was chosen to evaluate the bond strength, because of the small test area, what reduces the possibility of incorporating defects during the specimens preparation. Besides that, it allows the runnig of multiple test areas within a specimen, what reduces the material heterogeneity factor of the results.(28, 29). The performance of a shear test appears to be more clinically relevant, since components of tension, compression and shear are present as it occurs in clinical practice (30).

All composite surfaces were abraded with silicon carbide paper 150, 300 and 600 to obtain a surface roughness pattern (11, 13, 31, 32), since a filling material suffers drills action during tooth preparation for an indirect restoration. It has been speculated that roughening the surface, might remove the less reactive surface (13) and increase the surface roughness, which allows a micromechanical retention that enhance bond strength (5, 22, 33), corresponding as a high BS value to control group.

In this study, a total etch adhesive system was used for methacrylate-based composites and a corresponding self-etching system for the silorane-based composite, to the achievement of better compatibility between the filling and the composite resin cement. Indeed, some studies showed better bond strength values between methacrylate and silorane-based composites when the methacrylic phosphate adhesive system of P90 Adhesive System was used (21, 23, 32). It has been observed that the phosphoric acid acts just cleaning the surface (7, 34), it is not able to create micro retentions in composite surface (35, 36), which could higher the bond strength (26, 37) or not influence this property significantly(5, 26, 34, 38). However, a negative action was pointed by Kashi et al (39). Therefore, phosphoric acid application was done over all resin disks, regardless the type of material (silorane and methacrylate one).

Analyzing the results obtained, the hypothesis of the present study was accepted, whereas the bond strength values between a composite and a resin cement was dependent of the kind of composite and the surface treatment used. Indeed, avariability has been observed in how composites respond to a specific surface treatment, according to its chemical composition (10). Altogether, in the present study, the methacrylate-based composite showed higher bond strength values compared to silorane-based. In a previous work, most of the associations between a methacrylate and a silorane-based composite also showed lower bond strength values, compared to association between methacrylate-based composites (22). The association between silorane and methacrylate-based composites have been investigated and shown to be dependent of the intermediate agent used as a silane or an adhesive system (12, 21, 22, 32). The usage of Filtek LS Adhesive System, which contains as chemical base methacrylates monomers with addition of phosphate groups, have favored the bond strength values between silorane and methacrylate-based composites (21). The reaction between phosphate groups and oxirane and between acrylates groups of the adhesive and the dimethacrylate could be the responsible for this acceptable bond strength value (23). However, in the present study, the use of the adhesive system was not capable to turn the bond strength of Filtek LS similar to that obtained with the methacrylate composites. This can be observed comparing the result of control groups in all composites evaluated, according to previous study (22).

Generally, to sandblast with aluminium oxide shows higher bond strength values than only abrading the surface with silicon carbide paper. The superiority of this technique have been pointed out in previous studies (7-11, 25) and it is associated to a higher roughness and surface energy produced by sandblasting, what would allow a better adhesive flow in micro retention, improving the micromechanical retention between resin composite and cement. In this study the silica coating followed by silane application presented similar results to aluminum oxide sandblasting, which is in agreement to some other studies on composite bond strength (7, 13). On the other hand, some studies show differences between these two treatments depending on the type of composite evaluated (16, 27).

The ethanol application after sandblasting reduced the bond strength values for both methacrylic and silorane based composites. The longer time of application of ethanol used in this study, in comparision to previous ones (9), may have caused a solvent absorption by the composite, causing softening of the organic matrix, due to the remoteness from the chains of the polymer network (16, 40). But, the adhesive system monomers could not be able to infiltrate in the soft organic matrix, preventing a chemical bond. Also, it can be speculated that ethanol molecules were trapped in surface irregularities, influencing adversely the values of bond strength, as was pointed out in a previous study, which describes a polymerization inhibition of adhesive systems and composites by ethanol (33).

Evaluating the failure type, it can be observed (Table 2) that groups which had significantly lower bond strength, as P90-N, P90-SE, Z250-SE and Z3350-SE showed a higher number of adhesive failures and cohesive in cement. The higher amount of adhesive failures in these groups is in accordance with the low bond strength values between the substrate and the cement. The chisel-shaped tip, may have contributed to the cohesive failures cement present in all groups, due to a stress concentration in the cement cylinder (28). Nevertheless, this adversity related to the method was not significant due to the reduced amount of cohesive failure in cement, evaluating all experimental groups. As mechanical treatments, such as sandblasting and silica coating, promote an increase in surface roughness, the largest number of cohesive resin fractures for these treatments reflects the highest values of bond strength achieved, with the exception of the group P90 – C, that showed BS values lower than group control (N).

According to what were presented above, it can be observed that the use of surface treatments to increase the bond strength of resin materials is quite complex, with many variables results in the literature, since many factors can act concurrently. Thus, additional studies are needed in search better surface treatment protocols.

CONCLUSION

Within the limitations of this study and according to the results, ethanol treatment did not demonstrated appropriate values, and should not be indicated as a surface treatment.

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