Original Research Article

Is the bond strength of repairs influenced by the use of different bulk fill and nanoparticulate composites aged by acidic solutions that simulate the oral conditions of patients with bulimia nervosa?

Abstract

Purpose: This study aimed to evaluate the bond strength of aged and post-repair composites in different solutions, namely distilled water [DW]; 75% water/alcohol [WA]; and 0.02 N nitric acid [NA], which simulates the acidic conditions in the oral cavity of patients with bulimia nervosa. **Methods:** We used five composites: one nanoparticulate (Z350) and four bulk fills—Filtek Bulk Fill (FBF), Filtek Bulk Fill Flow (FBFF), Surefil SDR Flow (SURE), and Opus Bulk Fill Flow (OPUS). Samples were prepared from each material and aged for 30 days. The composites underwent a surface treatment followed by repair. The repaired samples were aged for another 30 days in the same solutions used previously and subjected to the tensile test with the universal test machine. The mean values obtained for each composite were analyzed using the Shapiro-Wilk and ANOVA tests, followed by the Tukey post-test ($p < 0.05$). **Results:** In most comparisons made among the composites, the nanoparticulate composite showed the worst bond strength values when used for repair, while the bulk fill composites showed the best results when used for repair. The highest bond strength values (MPa) were obtained with the combination of SURE/FBF-NA (8.41 ± 0.91), while the lowest values were obtained with the combination of SURE/FBFF-WA (1.57 ± 0.21). **Conclusions:** Combinations of different post-repair composites were better than those of the composites alone for most of the studied groups. The DW and NA solutions did not influence adhesive

strength. The WA solution reduced adhesive resistance in most studied combinations. The bulk fill composites showed higher adhesive resistance to the nanoparticulate composite for the repair of aging restorations. Aging in acidic solution affected the adhesive strength of repaired composites less than did aging in water/alcohol.

Keywords: aging solutions, bond strength, bulk fill, nanoparticulate composites, composite repair, bulimia nervosa

INTRODUCTION

Complete replacement of a composite restoration for aesthetic reasons is rather common, but in many cases, most of the portions removed are clinically and radiographically intact. Moreover, removal of the entire restoration is inevitably followed by wear of the healthy dental tissue and repeated pulp injuries [4, 11]. Therefore, repair of composite restorations is a conservative alternative for partial replacement of the infiltrated restorative material and allows preservation of part of the restoration and the healthy dental tissue [11]. Optimal bonding in the interface between the surfaces of the old restoration and the repair layer is a critical factor in such repair procedures, and the development of such bonding is reflected in the magnitude of the interfacial bond strength [26].

Studies on composite restorations have reported significant difficulties in establishing a durable bond between “aged” composites and the new composites used for repair [9]. The interaction between the two main factors, the type of composite and the surface treatment, is crucial in ensuring good bonding in composite repairs [2]. Although several *in vitro* studies have investigated the variables that affect the bond strength of repairs among composites, the bond strength required to achieve clinically satisfactory repair of a composite *in vivo* has never been assessed [22].

Different interactions of composites could be used to facilitate the repair technique. A rather interesting option is the use of bulk fill composites, although in cases with increments greater than 5 mm, manufacturers recommend the application of a superficial layer of composite to replace the occlusal or buccal enamel. Bulk fill composites appear to solve some of the disadvantages of conventional composites such as polymerization contraction and the consequent marginal microinfiltration. They also allow the use of an innovative system of polymerization initiation that involves an abbreviated light exposure time while increasing the

depth of cure of the bulk fill composites [12]. The low polymerization contraction and the high inorganic filler content (nanoparticles) reduce the contraction stress and allow the use of thicker layers than the 2-mm layers used conventionally [10].

Furthermore, composite restorations are exposed to the chemical agents found in saliva, foods, and beverages, which can accelerate composite degradation and increase unreacted monomer breakdown. These chemical agents may cause hydrolysis of the methacrylate ester bonds within the resin matrix of polymer-based materials [28]. The most frequently used solutions for material aging are water/alcohol and water; however, few reports have investigated the acid challenge faced by composites implanted in patients with bulimia nervosa [15], whose oral pH may reach 1.5. In this sense, the use of acidic aging solutions would simulate critical pH variations in the oral cavity; nitric acid could particularly simulate this condition because it has a pH close to 2.

Considering these conditions and the lack of studies verifying the viability of repairs using combinations of different types and brands of composites with aging using different solutions, this study aimed to assess the bond strengths of different combinations of post-repaired composites aged in different solutions.

METHODS

The sample size was calculated considering the probability distribution of the family F, with a repeated family design and interactions within and among the factors. The effect size used was 0.15, error type 1 (α) was 0.05, and analysis power was 0.90, which secured a minimum of 450 sample units (specimens), with six samples per experimental group. The sample size was calculated using GPower software (version 3.1.9.2 - University of Düsseldorf, Düsseldorf, Germany).

For this *in vitro* laboratory study, composite specimens (SPs) were produced by combinations of the five commercial brands assessed (Table 1) and stored in three different solutions—distilled water (DW), 70% distilled water/alcohol (WA), and 0.02 N nitric acid (NA).

The base of the SPs was created with a stick-shaped rubbery mold (ODEME) with original proportions (length × width × thickness) of 10 mm × 2 mm × 2 mm. This mold was divided longitudinally in half with a #15 scalpel blade (Swann Morton) without complete sectioning to avoid fully separating the parts. A polyester matrix (KDent, Quimidrol) was introduced in this region, and this crack was bonded with cyanoacrylate ester (Super Bonder, Loctite) so that the set would not move when the composite was inserted.

The composite was then inserted in half of the rubbery mold, and a polyester strip and a glass plate weighing 177 g were placed over it for 5 minutes; photoactivation was performed with a light-curing device (Blue Phase; Ivoclar Vivadent) with a power density of 1200 mW/cm^2, in accordance with the respective manufacturers' instructions. The light intensity provided by the photoactivator was verified using a radiometer (RD7; Ecel Indústria e Comércio Ltda) after every 10 uses. Thus, SPs were obtained with dimensions of 5 × 2 × 2 mm for the base.

Next, the SPs were stored for 30 days at 37°C in three different aging solutions (DW, WA, NA), which were changed every three days to maintain the pH of the solutions. The SPs were then dried and polished with a tile-colored and coarse soflex disc (Pop on/3M ESPE) in the margin such that the repair would be performed. In this margin, 37% phosphoric acid (Condac 37, FGM) was applied for 30 seconds, followed by washing for 30 seconds and drying with absorbent paper. The adhesive system (Single bond Universal; 3M ESPE) was applied and photoactivated with the light-curing device (Blue Phase; Ivoclar Vivadent) for 20 seconds, according to the manufacturer's instructions.

The SPs were returned to the rubbery mold with the treated part in the central region, and the remaining length of the SP (10.0 × 2.0 × 2.0 mm) was completed with the other composite to be assessed, in accordance with Figure 1. After filling the mold completely with composite, a polyester strip and a glass plate weighing 177 g were placed over it for 5 minutes, and photoactivation was performed with a light-curing device. After concluding the repairs, the SPs were immersed again in the aging solutions (DW, WA, NA) at 37°C for 30 days, and the solutions were changed every three days to maintain their pH.

For mechanical testing after the final storage, the SPs were bonded with cyanoacrylate adhesive gel (IC Gel, Bob Smith Industries) in a plastic bed for tensile tests (Tensile Jig Geraldeli 2; ODEME), and a standard metal jig (ODEME) was used to standardize the bonding position. The SPs were then coupled to a device for the tensile test in the universal testing machine (DL-200 MF; Emic/Instron Brasil, São Joé dos Pinhais, PR, Brazil) and subjected to the tensile test with load cell of 20 kg/F and speed of 0.5 mm/minute until the moment of fracture; these data were stored in a software package (Tesc; Emic). The fractured specimens were also examined with a stereomicroscope (SZX7a; Olympus) at 40x magnification by a single-blinded and calibrated operator to determine the failure mode, which was classified as adhesive, cohesive, or mixed, according to the criteria recommended by ISO TR 11405:1994 [13].

The data were subjected to the Shapiro-Wilk test, two-way ANOVA, and Tukey's test at a preset alpha of 0.05 using the Bioestat 5.3 software (Maua Institute, AM, Brazil).

RESULTS

Figures 2 through 6 present the results of the statistical analyses of the experimental groups. The Z350 base samples immersed in DW showed the lowest tensile strength when

repaired with Surefil (Figure 2). The highest value was observed in repairs performed with FBF, and repairing these samples with the same base composite did not yield the best results. When the samples were immersed in NA, the repairs performed with bulk fill flow composites were superior to those performed with Z350 or FBF composites (Figure 2). The FBF base samples immersed in DW showed the lowest tensile strength values when repaired with Z350, while the highest values were observed in repairs with Opus for the DW and NA solutions (Figure 3).

In the FBFF group, the highest mechanical strength values in samples immersed in DW and NA were observed when the repair was performed with the same commercial brand as the FBFF base, while other combinations showed mostly similar mechanical strength values among the possible combinations for aging in WA (Figure 4). Figure 5 shows similar mechanical strengths among all combinations after aging in WA and NA. In contrast, after aging in DW, the highest values were observed in repairs performed with Sure and Opus composites, while the lowest values were observed with Z350

In the Opus group, samples repaired with Z350 and FBF showed lower tensile strengths when immersed in DW and NA. When immersed in NA, repairs with Opus showed values superior to that obtained with FBFF and similar to that obtained with Surefil (Figure 6).

Tables 2 through 6 also present the results of the statistical analyses of the experimental groups. Analysis of the effect of different aging solutions on the tensile strength of the specimens produced with a Z350 base shows that the highest values were found in the groups immersed in DW, while the lowest values were observed for samples immersed in WA (Table 2).

Similarly, samples with an FBF base showed the lowest strength values when immersed in WA and repaired with the bulk fill flow composite. Although the mechanical

strength of the repairs in Z350 was not affected when immersed in different solutions, FBF base samples repaired with the same base composite presented the lowest values for immersion in WA, intermediate values for DW, and the highest values for NA (Table 3).

Table 4 shows that Z350, when used to repair samples with an FBFF base, was not significantly influenced by immersion in different solutions. In contrast, the other commercial brands behaved similarly, with the highest values observed with immersion in DW and NA and the lowest values with immersion in WA. The samples with a Surefil (Table 5) or Opus (Table 6) base presented similar behavior after immersion in the solutions assessed in this study. The lowest values were observed when the specimens were immersed in WA solution, and the highest values occurred after immersion in DW or NA.

Figures 7 through 9 present the frequency distribution of the types of failure found (%). They show that mixed failures occurred with all aging solutions and adhesive failures show the lowest occurrence. Cohesive failures in the WA solution (Figure 8) occurred most frequently for the Z350/Opus, Opus/Surefil, and Opus/FBFF combinations, and in the NA solution (Figure 9), this type of failure occurred for the Opus/FBFF and Opus/Surefil combinations. Adhesive failure was only prevalent in the NA solution (Figure 9) for the Z350/Z350 and FBFF/FBFF combinations.

DISCUSSION

The need for more conservative techniques to preserve the durability and viability of direct aesthetic restorations is increasingly becoming apparent. The present study assessed the bond strengths of different combinations of post-repaired composites aged in acidic solutions. The results indicated that composites from the same commercial brand do not always provide the highest bond strength values. Application of acidic solutions may also affect the bond strength values. This finding is rather interesting considering the general recommendation to

use the same material in repairs, based on past studies with conventional composites only. Moreover, studies have shown long-term problems such as recurrent caries and fractures in repairs using nanoparticulate composites [16,23]. Similar results were reported by Koc-Vural et al. [14], showing that the combination of bulk fill composites with conventional composites was superior to repairs with similar composites.

Assessments of multiple combinations of composite types and commercial brands showed that the Opus composite had significantly higher bond strength values in six combinations of results, that is, when immersed in NA solution with Z350, FBF, and Opus and when immersed in DW solution with FBF, Surefil, and Opus. This may be explained by the chemical composition of the Opus composite, which differs from the other composite because of the presence of the APS (advanced polymerization system) that may have facilitated these improved bond strength values [5]. Moreover, the load particle in Opus is predominantly composed of colloidal silica, which can form strong bonds with different organic matrices and shows lower toughness than zirconia and glass composites [8]. This may have facilitated the surface treatment with phosphoric acid, increasing surface irregularities and consequently improving the mechanical overlap of the adhesive system and composite, thus promoting higher bond strengths to the different materials tested, as observed in the present study.

In contrast, when used for simulated repairs, Z350 presented inferior results in the bond strength tests for eight of the combinations assessed. A similar result was found by Tavarez et al [27] who assessed the combination of Z350 with itself and with nanohybrid and bulk fill flow composites. These authors found the lowest micro-shear bond strength values in the combination of Z350 with bulk fill flow composites. Z350 is composed of 20-nm silica particles and 4–11-nm zirconia particles, while the other composites have nanohybrid and microhybrid compositions. The nanoparticles contain nano-agglomerates inside a matrix [17],

while the nanohybrid and microhybrid compositions involve the combination of two nano-agglomerates and conventional particles [6]. Moreover, the smallest size of the load particles may affect the bond strength results. An assessment of the mechanical and structural characteristics of the composite materials indicated a lack of studies assessing the post-repair bond strength [27].

Another factor contributing to the satisfactory results obtained with bulk fill composites was the modification of the organic matrix, which would increase translucency and reduce opacity, allowing for an increase in light transmission, and thereby ensure better polymerization and effectiveness of the mechanical properties. The photoinitiators in the bulk fill composites show higher initiation of free radicals than those in the conventional composites and allow the proper depth of cure over greater thicknesses [19], which may yield a superior degree of polymerization in bulk fill flow composites in comparison with Z350.

The use of NA for aging repaired samples is unprecedented in the literature, and this study sought to simulate the acidic conditions present in the oral cavity of patients with bulimia nervosa, an eating disorder characterized by episodic binge eating and inappropriate purging behaviors such as self-induced vomiting, laxative and diuretic misuse, and excessive exercise[15]. According to Smink et al. [24], eating disorders are a major public health concern with significant morbidity and mortality, and thus, it is of the utmost importance for dental studies to simulate the extreme oral conditions inherent in these disorders. Eating disorders can occur in the oral cavity, and professionals should be aware of these situations so that they can customize the composite restorations or repairs accordingly. In the present study, the low pH found in patients with eating disorders was simulated by immersing samples in NA; despite this low pH, similar adhesive resistance values were unexpectedly observed for the NA and DW solutions. Backer et al. [3] have verified that composites can resist microhardness degradation; in the study by Zaki et al. [30] the Z350 composite presented

similar microhardness values in the non-immediate periods (aging of 6 and 12 hours). Thus, the findings of the present study appear to be justified by the ability of modern composites to resist the action of acid solutions. Further, 30-day storage in water can provide an aged composite surface [21] with reduced radical activity of monomer functional groups. In this sense, the aging protocol for composites used in the present experimental design may have yielded conditions resulting in similar adhesive resistance between the DW and NA groups.

In contrast to the findings for DW and NA, storage in WA solution significantly reduced the bond strength values for all composite combinations. Yesilyurt et al. [29] and Mohammadi et al. [18] also reported significantly lower flexural strengths after storage in WA. A previous study investigated the aging process by verifying the effects of oral fluids on the stability of composite materials by exposing the composites to water, artificial saliva, and WA solutions with different concentrations, including 75% and 50% [28]. The effects of these chemical compounds are different, but typically include leaching of unreacted monomer components and a destructive effect on the polymer network. The organic solvents absorbed by the polymer network of the methacrylate-based composites form a small percentage of their total weight. This network does not dissolve because attractive forces between polymer chains are stronger than those between solvent molecules and components of the polymer chain [7.28]. Thus, variations from the interactions of secondary monomers will lead to an increased network volume, causing higher polymer plasticity. Simultaneously, the polymer chains will be separated by molecules that do not show primary chemical connections to the chain but influence the structure by the spatial occupation of the polymer network. Therefore, the main effect of the solvent is a reduction in the interactions among polymer chains. Plasticity is proportional to the amount of solvent absorption, which starts immediately and reaches its maximum rate within one or two months, when the network is completely saturated by the solvent. In a study by Vouvoudi & Sideridou [28], a 75% aqueous solution of

ethanol had a higher effect on the mechanical properties of the composites than water and artificial saliva, which may be attributable to the organophilic nature of ethanol. Ethanol causes softening and degradation of the polymer matrix and eliminates the connection between filler particles and organic matrix.

Analysis of the type of failure may aid interpretations of bond strength results, as in the present study. The bulk fill flow composites (FBFF, Opus, Surefil, and their combinations) showed cohesive failures, which indicated that the bond strength of the adhesive interface was higher than the cohesive strength of the material. Similar results were found by Subaşı & Alp [25] and Altinci et al. [1]. The remaining combinations showed mixed failures more often, as reported by Ozdemir et al. [20] who noted failures between the porcelain and the composite. In contrast, the FBFF/FBFF and Z350/Z350 combinations in NA solution showed adhesive failure more often, matching the results for the intermediate bond strength.

Although laboratory tests cannot accurately reproduce clinical conditions, they are a major analysis parameter, because efficient *in vitro* performance may result in improved clinical performance. Thus, when extrapolating the results of the present study for the *in vivo* condition, it should be considered that for composite restoration repairs, the best option would be the use of bulk fill composites instead of conventional nanoparticulate composites.

CONCLUSIONS

The results suggest that for most of the groups studied, combinations of different composites present better results than combinations of similar composites. The DW and NA solutions did not affect the bond strength pattern. The WA solution reduced the bond strength for most of the combinations studied.

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Figure legends

Figure 1 Sequence of production and distribution of the specimens in the experimental groups.

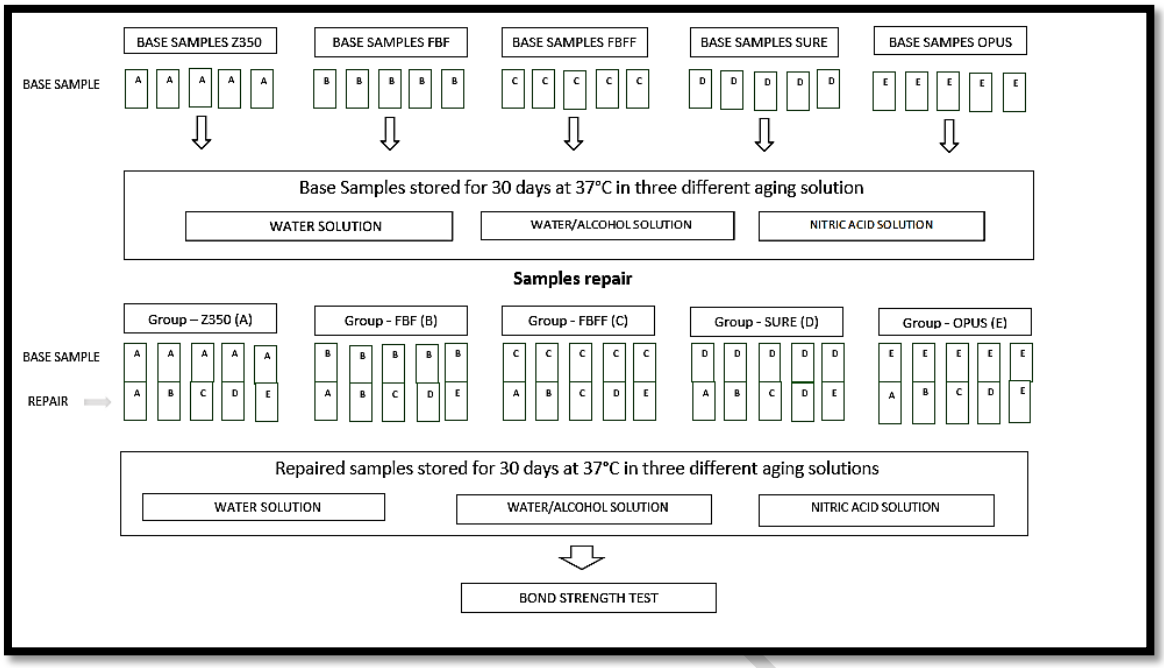

Figure 2 Bond strengths in the tensile tests with the Z350 base samples in the experimental groups, analysis intrasolution (MPa).

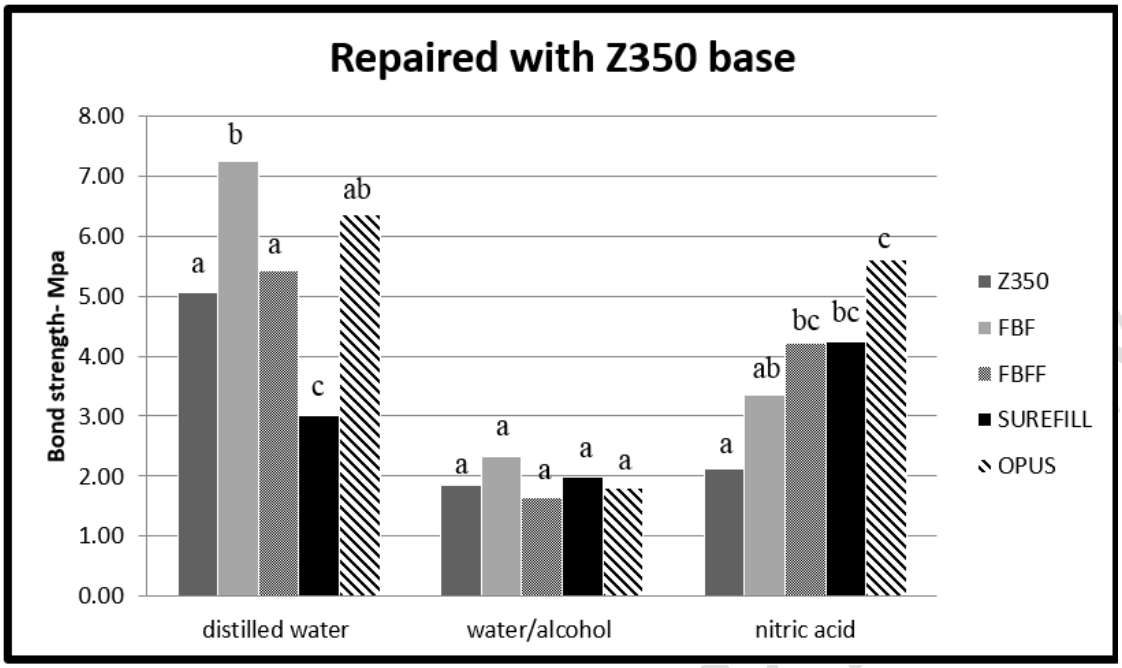

Figure 3 Bond strengths in the tensile tests with the Filtek Bulk base samples in the experimental groups, analysis intrasolution (MPa).

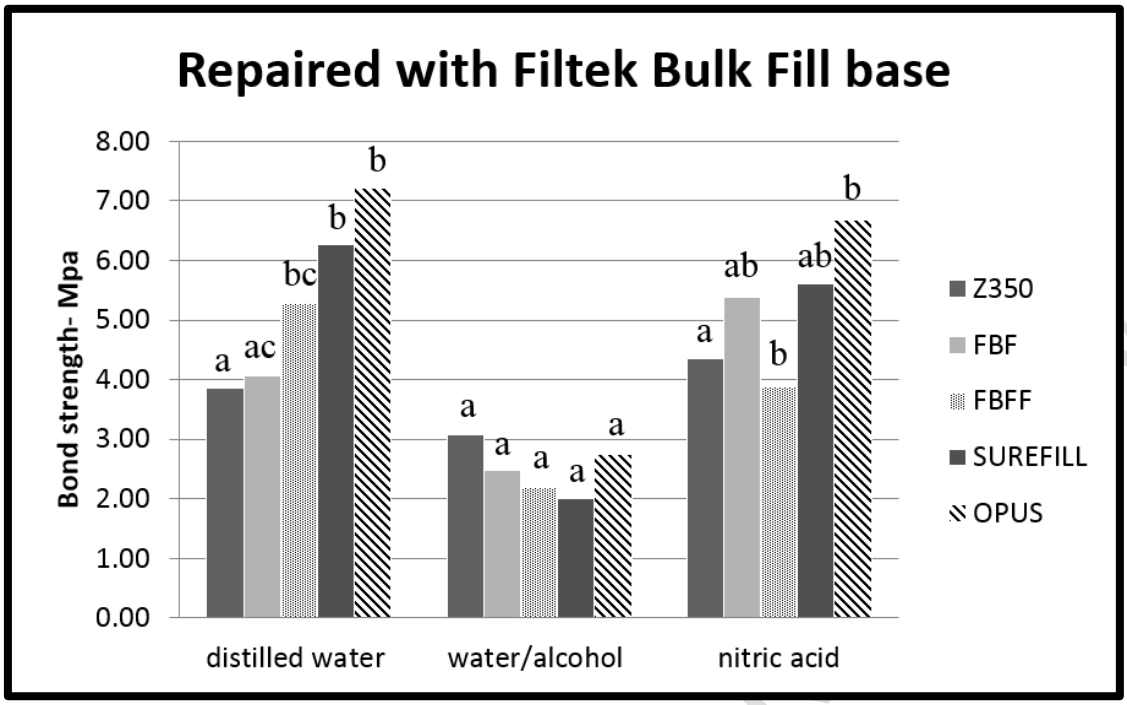

Figure 4 Bond strengths in the tensile tests with the Filtek Bulk Fill Flow base samples in the experimental groups, analysis intrasolution (MPa).

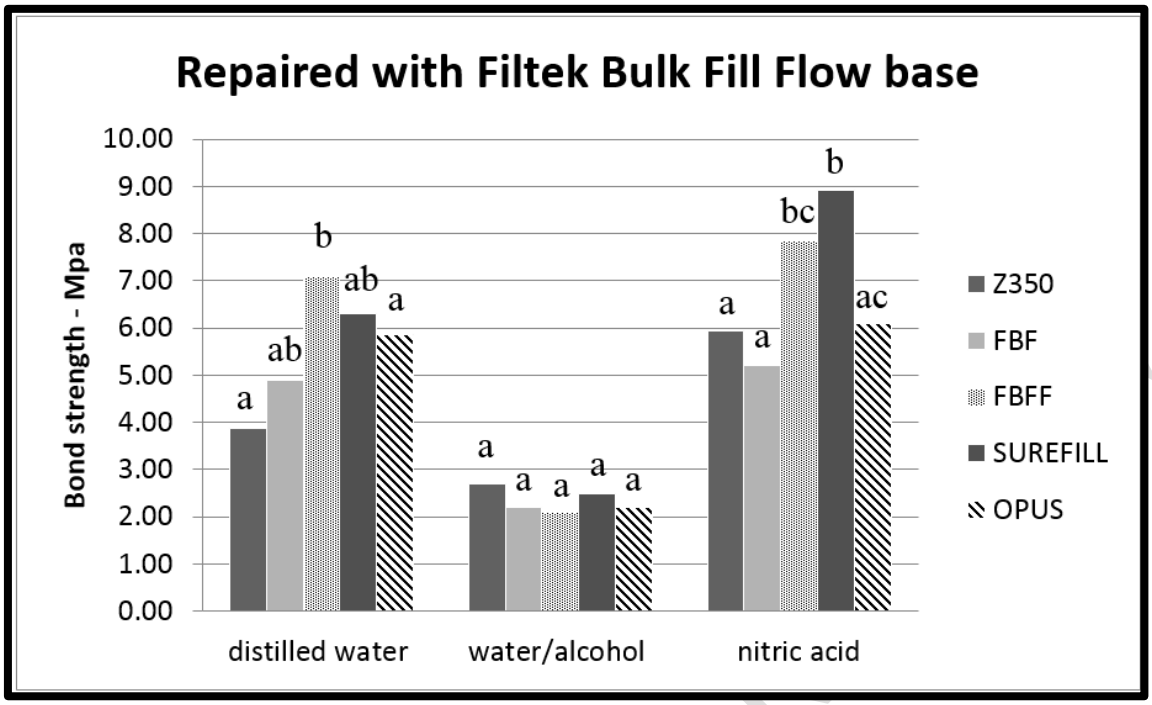

Figure 5 Bond strengths in the tensile tests with the Surefil Fill SDR Flow base samples in the experimental groups, analysis intrasolution (MPa).

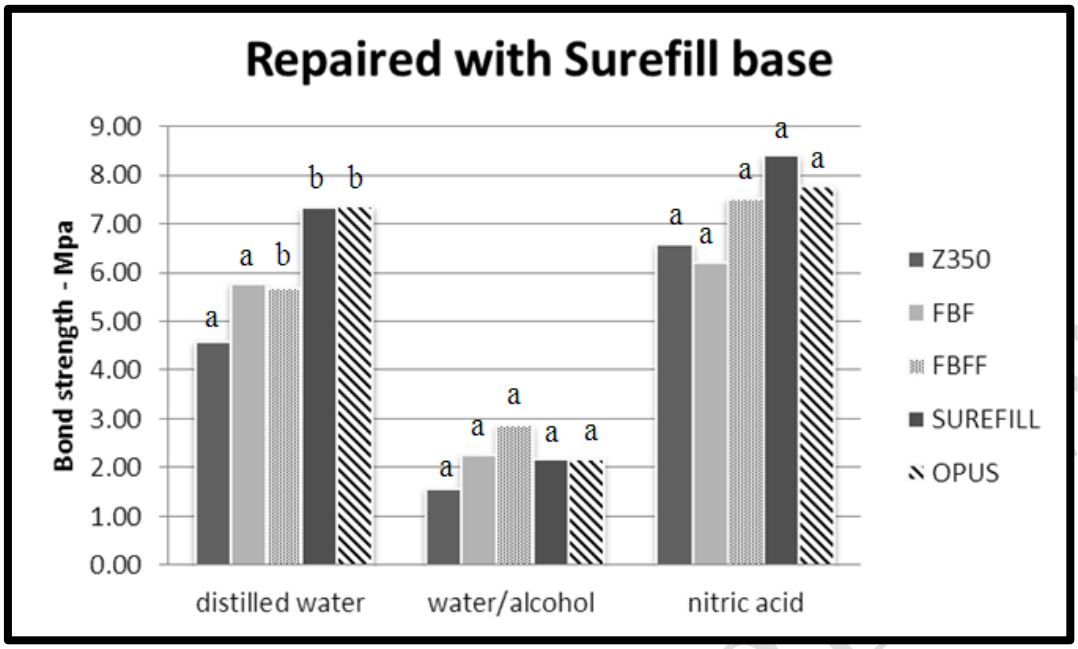

Figure 6 Bond strengths in the tensile tests with the Opus Bulk Fill base samples in the experimental groups, analysis intrasolution (MPa).

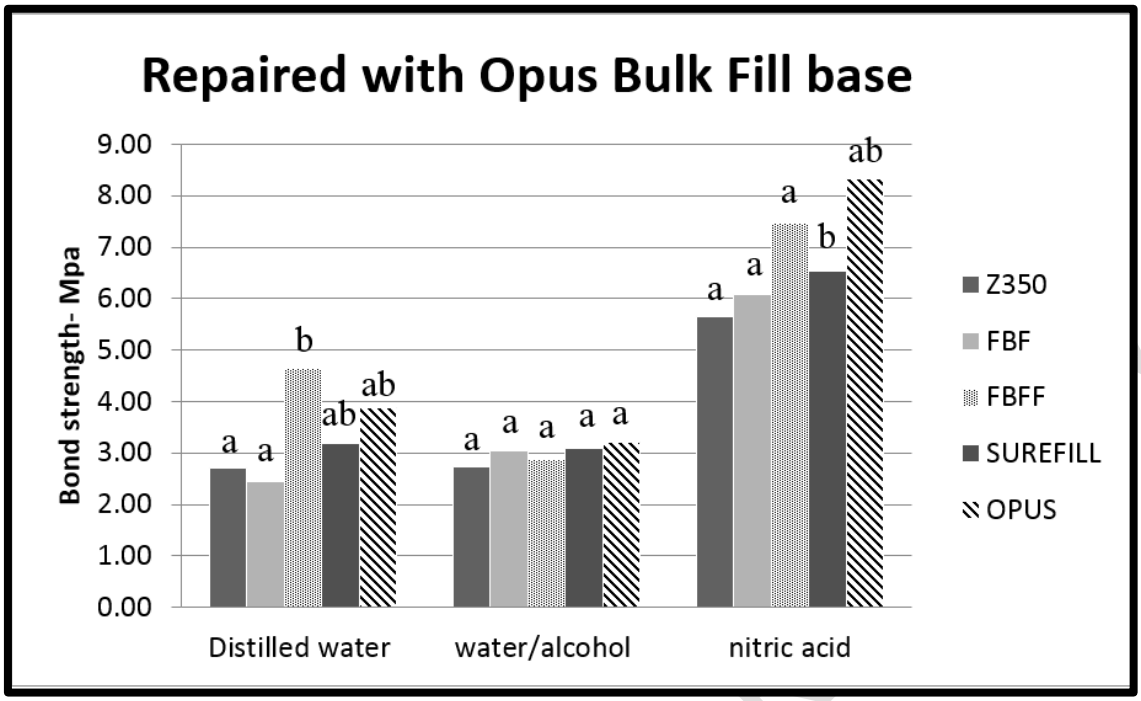

Figure 7 Frequency distributions of the failures observed in the experimental groups according to the base composite aged in distilled water solution.

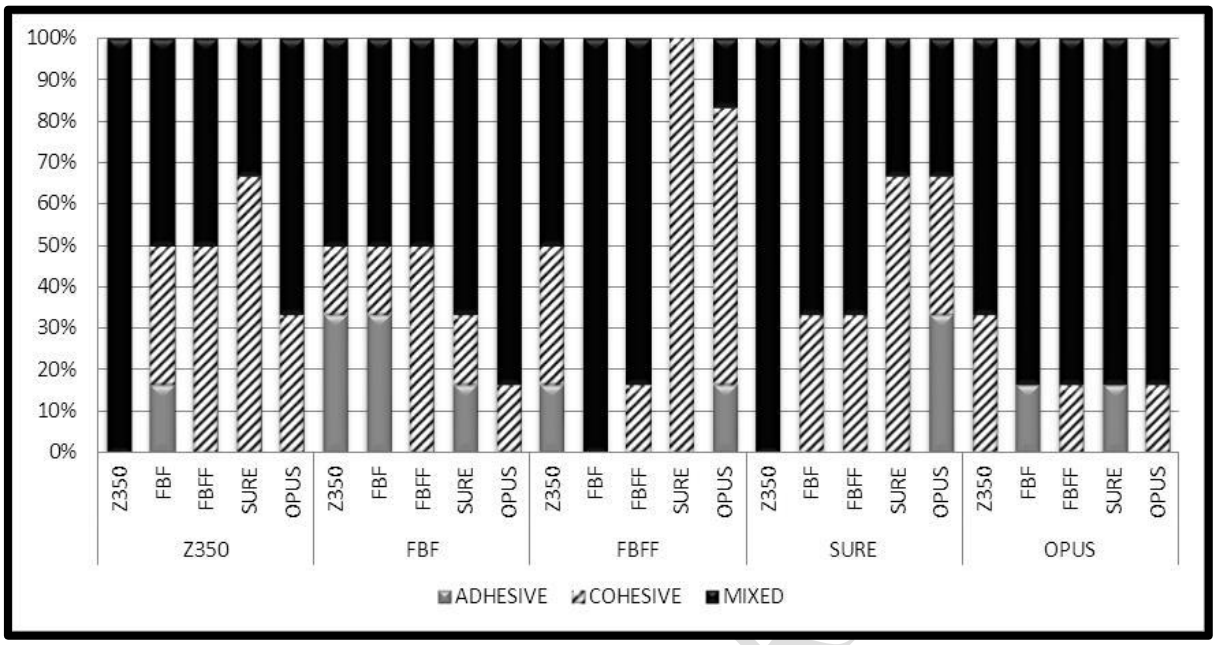

Figure 8 Frequency distributions of the failures observed in the experimental groups according to the base composite aged in water/alcohol solution.

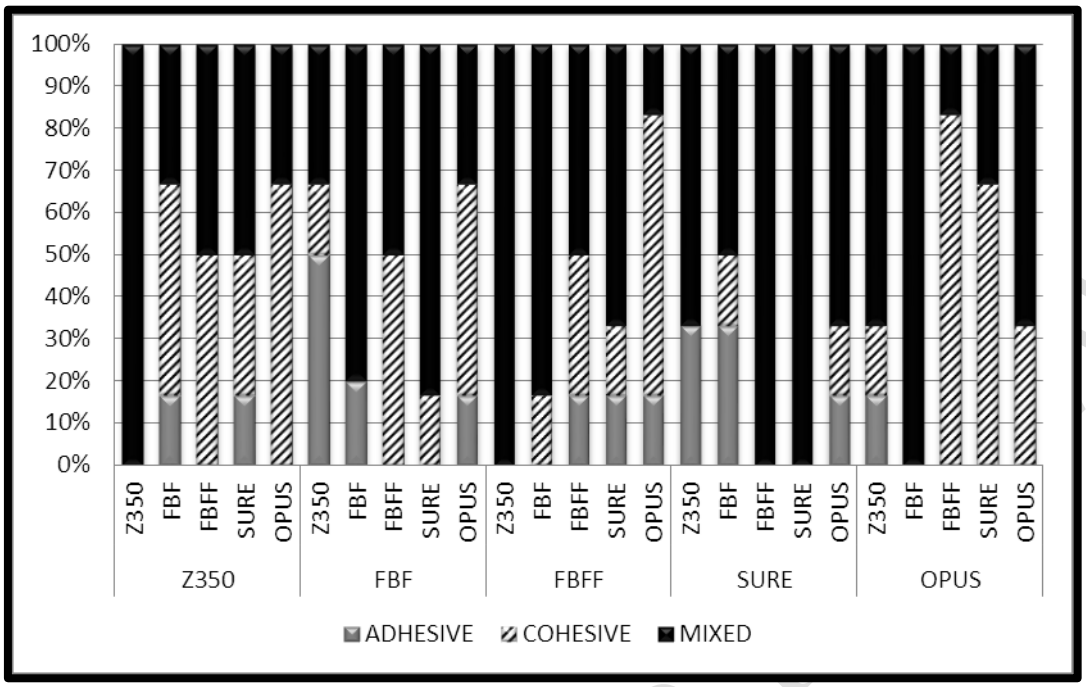

Figure 9 Frequency distributions of the failures observed in the experimental groups according to the base composite aged in nitric acid solution.