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Effect of Intermediate Agents and Pre-heating of Repairing Resin on Composite-repair Bonds

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Clinical Relevance

The combination of a flowable composite as an intermediate agent and a repairing material at a temperature of 23°C or 37°C prior to light curing may be recommended as a simple, suitable procedure to provide higher bond strengths and uniform composite-to-composite interfacial adaptation.

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SUMMARY

This study investigated the composite-to-composite microtensile bond strength and interfacial quality after using different combinations of intermediate agents and pre-curing temperatures of repairing resin. Forty-five composite discs (8x4mm) of Gradia Direct Anterior (GC Corp), stored in a saline solution at 37°C for one month, were sandblasted (50 µm aluminum oxide), cleaned (35% phosphoric acid) and randomly divided into three groups (n=15) according to the intermediate agent applied: 1) no treatment; 2) unfilled resin (Scotchbond Multi-Purpose Adhesive, 3M ESPE); 3) flowable composite (Gradia LoFlo, GC Corp). Each disc was incrementally repaired (8x8 mm) with the same resin as the substrate. For each group, three subgroups (n=5) were created, depending on the pre-curing temperature of the repairing resin—4°C, 23°C or 37°C. Two bonded specimens per group were prepared to evaluate the composite-to-composite interfacial quality via scanning electron microscope. Microtensile bond strength measurements were performed with

the remaining three specimens and failure mode was examined by stereomicroscopy. Two-way ANOVA revealed that temperature ($p<0.001$), intermediate agent ($p<0.001$) and the interaction ($p=0.002$) significantly affected the repair strength. Post-hoc comparisons revealed that bond strengths were significantly lower using a 4°C repairing resin in groups where intermediate agents were used. The highest bond strengths were recorded when flowable composite was used as an intermediate agent under each of the three temperature conditions. Interfacial quality improved by raising the resin temperature from 4°C to 37°C.

INTRODUCTION

In the last decade, growing demands by patients for mercury-free, esthetic restorations has markedly increased the use of direct, light-activated resin composites in restorative dentistry.[1] However, despite the continuing development of resin composites with improved properties, several factors, such as discoloration, color mismatch, wear, chipping or bulk fracture may still present concerns.[2-4] As a result, when esthetics or function are compromised, an operative treatment is needed, and the clinician must decide whether to replace or simply repair these restorations. When failure is limited to resin composite bulk and the tooth-resin interface is free of staining or secondary caries, replacement of the whole restoration would unnecessarily involve more complex, costly treatments, both in economic and biological terms.[2] Minimally invasive approaches, such as selective composite repair or refurbishing, should be the preferred method.[5]

In order to achieve long-term clinical success, an intimate resin-to-resin coupling[6] and high interfacial strength are required when repairing a composite restoration. Due to the limited amount of residual free-radicals available for reacting with new resin monomers on the old, contaminated, highly cross-linked resin matrix of composite substrate,[7-9] chemical and/or mechanical treatments have been investigated for preparation of the surface to be repaired.[10-15] Aluminum oxide sandblasting, followed by application of a bonding agent, proved to be a reliable method for enhancing composite repair strength.[8,12,15] Benefits in resin-to-resin bond seem to arise from the physico-chemical changes induced by the air-abrasion process. This results in a roughened composite surface, thus improving the surface area available for bonding.[6,8] In addition, an increase in surface energy is claimed to facilitate wettability of the intermediate agent,[16] thus promoting a closer adaptation to the composite substrate.[8,10,13] As a consequence of the penetration of resin monomers into surface irregularities, a micromechanical interlocking is achieved.

Filled[8,17-18] and unfilled[6,9-13,19-21] resins usually have been applied as intermediate agents for composite repair, thus achieving comparable results in terms of interfacial quality and strength.[17-18] Recently, the application of flowable composites as intermediate agents with[19-20] or without[18] a preliminary adhesive coating was demonstrated to be a valid alternative to traditional bonding procedures for composite repair.

Resin composites are traditionally stored at temperatures ranging from 2°C to 25°C, as suggested by manufacturers, and retrieved from the storage place immediately prior to use. As resin viscosity is temperature-dependent and directly related to the handling characteristics of the composite,[22-24] differences in ease of placement and shaping on the repaired site could be expected in a clinical situation, potentially affecting interfacial quality and repair strength.

This study evaluated the effect of different pre-curing temperatures of repairing resin on composite-to-composite bond strength and interfacial micromorphology. The tested null hypothesis is that different combinations of intermediate agents and storage temperatures of the repairing resin do not affect the adhesion between an aged composite substrate and a fresh composite material.

METHODS AND MATERIALS

Forty-five cylinder-shaped composite discs were made by layering 1 mm thick increments of a micro-filled hybrid resin composite (Gradia Direct Anterior, GC Corp, Tokyo, Japan, shade A3) in a split aluminum mold (8 mm deep x 8 mm in diameter). An internal piston was adjusted to a depth of 4 mm in order to standardize the height of the composite discs. Each increment was condensed with a clean plastic filling instrument to avoid contamination and light-cured for 20 seconds (VIP, BISCO Inc, Schaumburg, IL, USA, output: 600 mW/cm^2). The last increment was covered with a Mylar strip and compressed with a glass microscope slide in order to obtain a flat surface. Each specimen was removed from the mold, additionally light-cured on the portions previously in contact with the metallic surface of the mold and stored in saline solution at 37°C for one month. One flat composite surface of the cylinder was selected, then sandblasted with 50 µm aluminum oxide powder for 10 seconds at a working distance of 5 mm at a pressure of $5.5 \cdot 10^5$ Pascals (Pa) (Microetcher II, Danville Engineering Inc, San Ramon, CA, USA). For purposes of cleaning, a 35% phosphoric acid etchant (Scotchbond etchant, 3M ESPE, St Paul, MN, USA) was applied for 30 seconds. After water rinsing and air drying, composite discs were randomly divided into three experimental groups (n=15) according to the different intermediate agents used before applying the repairing resin: Group 1: direct bonding between old and fresh composite; Group 2: application of an unfilled bonding agent (Scotchbond Multi-Purpose Adhesive, 3M ESPE); Group 3: application of a low-viscosity flowable composite (Gradia LoFlo, GC Corp). The tested intermediate agents were stored and used at room temperature (23°C).

For each experimental group, mono-dose tips containing the uncured repairing resin (Gradia Direct Anterior) were stored at different temperatures before use: 4°C (refrigerator); 23°C (room temperature); 37°C (incubator), leading to three subgroups (n=5).

Each specimen was returned to the mold, and a new restoration was packed onto the previously treated surface by layering 1 mm thick increments of the repairing resin and obtaining an 8 mm high cylindrical repaired sample. The repairing material was the same as the respective composite substrate except for its shade (Gradia Direct Anterior, shade B3). This was intentionally done to facilitate the positioning of the repair interface of microtensile specimens in the testing machine and to visually identify the failure site, particularly when no intermediate agent was applied.

All the materials were used according to manufacturers' instructions. Batch numbers, application mode and chemical composition are reported in Table 1.

Composite Repair Microtensile Testing

After a 24 hour-storage in saline solution at 37°C, three bonded specimens from each subgroup were randomly selected and cut with a slow-speed diamond saw under water cooling (Isomet, Buehler, Lake Bluff, IL, USA) to produce multiple beam-shaped sticks with a cross-sectional area ranging between 0.79 mm^2 and 0.81 mm^2 according to the non-trimming technique of the microtensile bond strength test.[25] The sticks consisted of the old composite in one-half of their length and the new one in the remaining half. Each beam was glued at the ends of a Gerardeli's jig[26] with cyanoacrylate glue (Zapit, Dental Ventures of America, Inc, Corona, CA, USA) and loaded in tension at a crosshead speed of 0.5 mm/minute in a universal testing machine (Controls, Milano, Italy). The load at failure was recorded in Newtons (N). The cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital caliper in order to express bond strength values in MegaPascals (MPa). The fractured beams were evaluated under a stereomicroscope (SMZ645, Nikon Co, Tokyo, Japan) at 50x magnification to record failure modes, classified as cohesive (within the old/repairing resin composite), adhesive (at intermediate agent/resin composite interface) or mixed (a combination of cohesive/adhesive failure modes). When no intermediate agent was applied, adhesive failure was considered to be the fracture occurring at the composite-to-composite interface. The number of sticks representative of the three failure patterns within each experimental subgroup was expressed as a percentage.

Statistical Analysis

All the sticks that failed prematurely (two sticks for no intermediate agent/4°C subgroup and one stick for Scotchbond Multi-Purpose/4°C subgroup) were considered as "zero bond" values and were included in the statistical calculations. After analyzing the bond strength data for normality of data distribution (Kolmogorov-Smirnov test) and homogeneity of variances (Levene's test), a two-way ANOVA was applied, with bond strength as the dependent variable, intermediate agent and repairing resin temperature as factors. The Tukey test was used for post-hoc multiple comparisons. All analyses were processed using SPSS 11.0 software (SPSS, Chicago, IL, USA). The level of significance was set at $p<0.05$.

Scanning Electron Microscopy (SEM) Evaluation

The two remaining bonded specimens in each subgroup were processed for composite-to-composite interfacial

Table 1: *Batch Numbers, Composition and Application Mode of the Materials Tested in the Study*

Material	Batch #	Composition	Application Mode
Gradia Direct Anterior (GC Corp, Tokyo, Japan)	A3: 0501121 B3: 0501191	*Matrix:* UDMA, dimethacrylate co-monomers *Filler*: silica (22 vol%), prepolymerized fillers (0.85 µm, 42 vol%) Pigments, catalysts	Apply a 1 mm thick layer. Light-cure for 20 seconds.
Scotchbond Etchant (3M ESPE, St Paul, MN, USA)	5FM	35% H_3PO_4	Apply for 30 seconds. Water-rinse. Air-dry.
Scotchbond Multi-Purpose Adhesive (3M ESPE, St Paul, MN, USA)	3NM	Bis-GMA, HEMA, photoinitiator	Apply a thin layer. Light-cure for 20 seconds.
Gradia Direct LoFlo (GC Corp, Tokyo, Japan)	0505301	*Matrix*: UDMA, multifunctional methacrylate monomers. *Filler*: HDR pre-polymerized fillers (20 µm), fluoro-alumino silicate glass (1.7 µm), nano-silica fillers (7 nm).	Apply a thin layer. Light-cure for 20 seconds

Abbreviations: Bis-GMA: Bisphenol A-diglycidylmethacrylate; HDR: high-density radiopaque; H_3PO_4: phosphoric acid; HEMA: Hydroxy ethilmethacrylate; UDMA: Urethane dimethacrylate.

characterization. Each specimen was cut perpendicular to the bonded interface with a slow-speed saw under water cooling (Isomet, Buehler), resulting in five 1 mm thick slabs. Each slab was then polished with SiC papers of increasing grit (#600, #1000, #1200, Buehler), rinsed with deionized water, air-dried, mounted on aluminum stubs and sputter-coated with gold (Polaron Range SC7620, Quorum Technology, Newhaven, England). The samples were observed under a scanning electron microscope (JSM 6060 LV, JEOL, Tokyo, Japan) at different magnifications.

RESULTS

Composite-to-Composite Microtensile Bond Strength

The results of microtensile bond strength testing for each experimental group are summarized in Table 2. Two-way ANOVA revealed that the temperature ($p<0.001$), the intermediate agent ($p<0.001$) and the interaction between these two factors ($p=0.002$) significantly affected composite repair strength. Bond strengths were significantly lower, using a 4°C-repairing resin in groups where intermediate agents were not used. The highest results were recorded when flowable composite was used as an intermediate agent under each of the three temperature conditions of the repairing resin. In groups where no intermediate agent was used, bond strengths were the lowest when using a 4°C- and a 23°C-repairing resin. However, when Gradia Direct Anterior was preheated at 37°C, statistically superior values were achieved, which were comparable to bond strengths in adhesive-mediated groups.

Stereomicroscopy and SEM Analysis

The distribution of failure modes in the tested groups is reported in Figure 1. Interfacial failure at the composite-to-composite interface mainly occurred when no intermediate agent was applied, regardless of the precuring temperature of the repairing material. Cohesive and mixed failures were mostly recorded when Scotchbond Multi-Purpose was used. Adhesive and mixed failures were predominant in the Gradia Direct LoFlo/4°C subgroup, while a frank cohesive failure in substrate/repairing resin was the main failure mode when Gradia Direct

Table 2: *Microtensile Bond Strength of the Tested Groups*

		Temperature		
	Intermediate Agent	4°C	23°C	37°C
1.	No intermediate agent	25.4 ± 6.7^{EF} [28]	24.5 ± 5.2^{F} [32]	35.4 ± 7.7^{CD} [33]
2.	Scotchbond Multi-Purpose Adhesive	30.0 ± 7.9^{DE} [34]	38.2 ± 6.4^{C} [31]	40.7 ± 6.2^{BC} [33]
3.	Gradia LoFlo	36.8 ± 7.3^{C} [33]	43.8 ± 6.9^{AB} [30]	48.1 ± 6.8^{A} [28]

Values are mean (± standard deviation) in MPa. The number of specimens tested for each group is reported in square brackets. Groups identified by different superscript letters are statistically dissimilar (p<0.05).

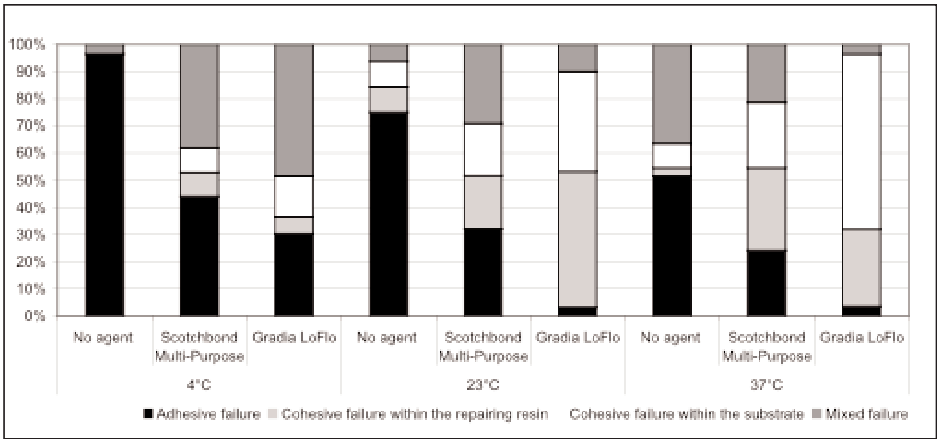

Figure 1: *Distribution of failure modes for all experimental groups. Values are the percentage of sticks fractured in the respective failure mode within each subgroup.*

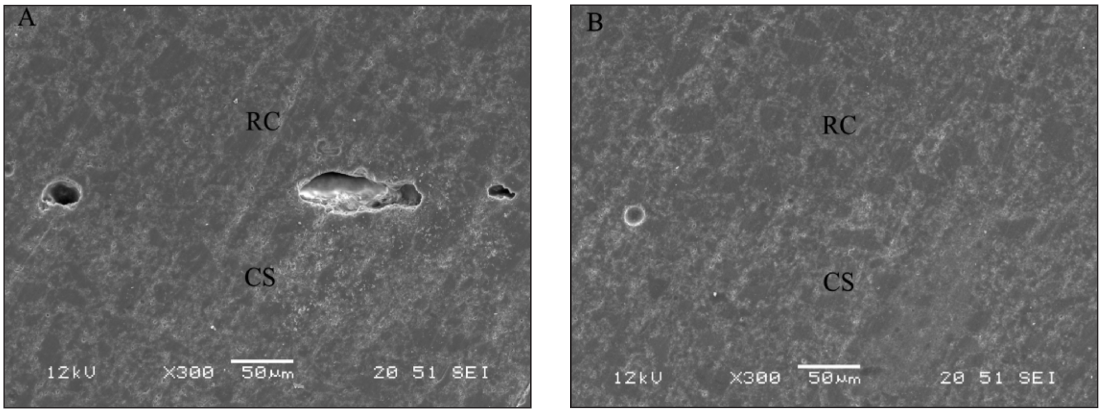

Figure 2: *Representative Gradia-to-Gradia interfaces repaired by direct bonding of the repairing composite (RC) to the sandblasted composite substrate (CS) (bar=50 µm). Figure 2A. SEM micrograph of CS repaired by using 4°C-cooled RC. Gaps (20-100 µm) and discontinuities interrupt the interfacial coupling. Figure 2B. SEM micrograph of CS repaired with 37°C pre-heated RC. A good interfacial composite-to-composite adaptation barely interrupted by small defect (less than 15 µm) is detectable.*

LoFlo was covered by 23°C- and 37°C-composite. No failures occurred within the flowable resin.

When direct composite-to-composite coupling was performed, SEM evaluation of bonded interfaces showed an effect of resin temperature on interfacial quality. Gaps and defects decreased by increasing the repairing resin temperature from 4°C to 37°C (Figure 2A,B). When Scotchbond Multi-Purpose was used as the intermediate agent, interfacial defects visible at the adhesive/4°C-composite interface (Figure 3A) disappeared by layering 23°C- and 37°C-composite, showing a better interfacial adaptation (Figure 3B). While layering 4°C-cooled composite over flowable Gradia LoFlo resulted in imperfect coupling (Figure 4A), the use of 23°C- and 37°C-composite allowed for a more uniform, complete composite-to-composite adaptation (Figure 4B).

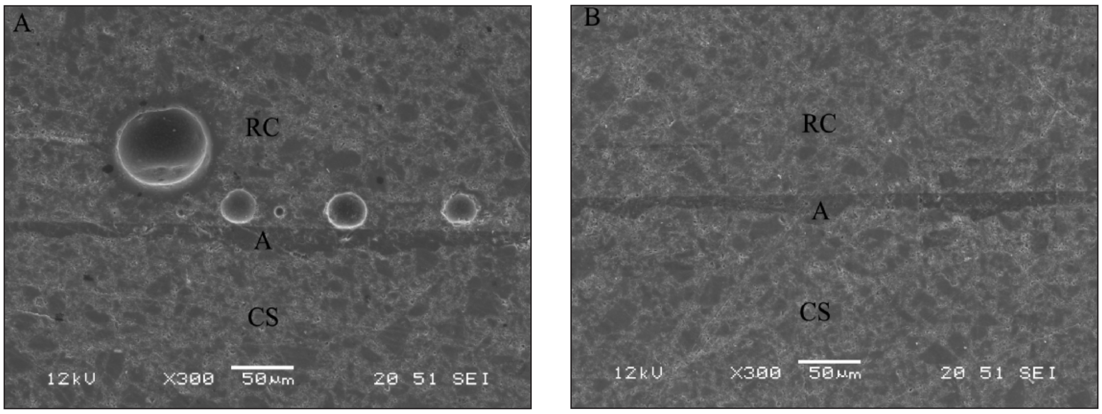

Figure 3: *Representative Gradia-to-Gradia interfaces repaired by unfilled resin Scotchbond Multi-Purpose Adhesive as an intermediate agent (RC: repairing composite adhesive resin, CS: composite substrate) (bar=50 µm). Figure 3A: Voids and defects (10-70 µm) both in the RC and in close proximity of the adhesive layer (A) are detectable with 4°C-RC. Figure 3B. A uniform adaptation at the CS-A and A-RC interfaces are evident with 23°C-RC.*

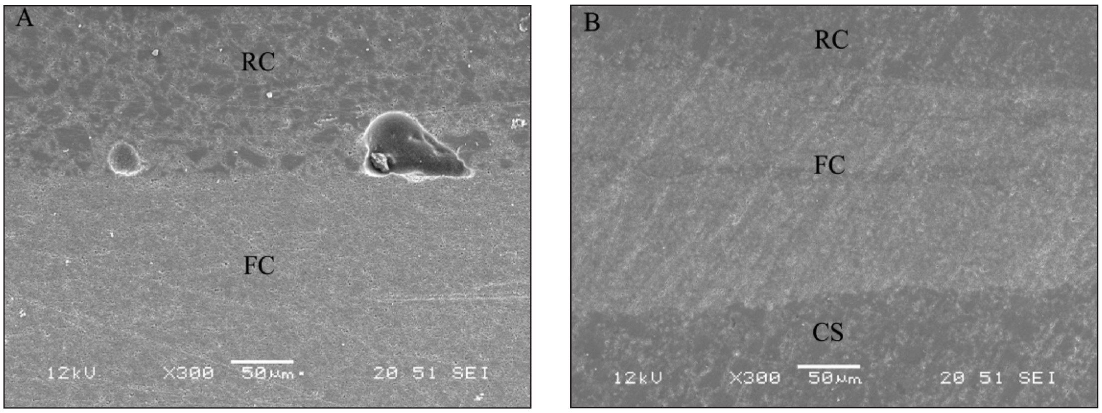

Figure 4: *Representative Gradia-to-Gradia interfaces repaired by flowable composite Gradia LoFlo as an intermediate agent (RC: repairing composite, FC: flowable composite, CS: composite substrate) (bar=50 µm). Fiigure 4A. Particular of 4°C RC-FC interface. A gap (50-100 µm), probably resulting from difficult adaptation of RC is clearly shown. Figure 4B. 23°C-composite as RC. Good coupling at both CS-FC and FC-RC interfaces is visible.*

DISCUSSION

Significant differences in repair strength were found between the experimental groups, depending on the intermediate agent and composite temperature preceding photo-polymerization. When groups in which the repairing resin temperature was the same were compared, the highest composite-to-composite strengths were achieved using a flowable composite as an intermediate agent under each of the three temperature conditions. As the interaction between intermediate agent and temperature was significant, the effect of the repairing resin temperature depended on the intermediate agent applied and vice versa. Hence, the null hypothesis tested has to be rejected.

Rheology studies indicated that resin composites, being visco-elastic materials by nature, respond to an increase in external temperature according to the Arrhenius equation, which results in the exponential decrease in their viscosity.[24,27-28] A decline in viscosity of the repairing resin, achieved by raising the temperature from 4°C to 37°C, resulted in a critical factor in improving composite-to-composite interfacial coupling and strength.

A composite repair, based on direct bonding between the repairing resin and the sandblasted substrate, is not usually recommended. There are two factors that may impair adhesion: the low chemical bonding potential of the aged substrate[7-9] and the incomplete penetration of the highly viscous fresh composite into surface pits and depressions.[13,21] Thus, the application of a bonding resin as intermediate agent is advisable for enhancing substrate wetting.[10,12] In this study, similar repair strength values were obtained by using cooled and room-temperature-stored composite as repair materials without applying any intermediate agent. However, in spite of comparable bond strength results, SEM analysis demonstrated an inferior composite-to-composite adaptation with 4°C-composite. Clear discontinuities could be more frequently observed compared to interfaces formed using 23°C-composite.

Conversely, superior repair strengths and a complete, more uniform coupling with the substrate, were achieved by warming the repairing resin composite to 37°C. The increased temperature from 23°C to 37°C presumably allowed the repairing composite to achieve optimal viscosity and flowing properties, thus promoting a deeper resin interdiffusion into surface microretentions. A significant improvement in repair strength was also achieved. The prevalence of adhesive failures with 4°C-composite (95%) and their progressive reduction with 23°C- (75%) and 37°C-composite (55%) seemed to confirm the beneficial contribution of pre-heated, low-viscosity repairing material to micromechanical retention. It is interesting to note that bond strengths achieved by direct bonding of the pre-heated resin were similar to those attained by applying the unfilled resin Scotchbond Multi-Purpose Adhesive as intermediate agent, regardless of the precuring temperature. However, the smaller percentage of adhesive failures recorded with Scotchbond Multi-Purpose (45% for 4°C-, 30% for 23°C- and 25% for 37°C-composite) may reveal the formation of a bonded interface that is more prone to withstand tensile stresses. Moreover, since composite-to-composite bond strength was higher when flowable resin was used at 23°C, composite repair without the use of an intermediate agent should not be routinely recommended, even if the repairing composite is warmed at 37°C.

A noteworthy finding of this investigation was the statistically superior performance of Gradia LoFlo as an intermediate agent. Since flowable composites possess lower mechanical and physical properties than traditional hybrid materials,[29-31] their application in occlusal, high stress-bearing areas could represent a concern. Therefore, the use of flowables as intermediate agents prior to layering a repairing hybrid composite has been proposed.[18-20] This allows flowable composites to overcome their potential clinical limitations, taking advantage of their stress-relieving behavior.[32-33] Whereas adhesive coating preceded the application of flowable composite as an intermediate agent in some studies,[19-20] a flowable resin was used as the sole interfacial material in a recent investigation.[18] It may be argued that the high polymerization shrinkage of flowable composites could generate high interfacial stress,[34] possibly impairing the composite-to-composite bond. However, no gaps or discontinuities were observed at the composite substrate-flowable intermediate interface. This may depend on the low intrinsic rigidity traditionally exhibited by flowable materials when compared to unfilled resins.[34] As a consequence of their counteracting role, the interfacial integrity may be preserved.[32-33] The higher thickness of the flowable intermediate agent (about 200 µm) to that of the unfilled resin (about 25 µm) is an additional factor that may have improved the stress-relieving potential of the composite-repair joint.[33-35] The predominance of cohesive failures within the substrate/repairing material, instead of adhesive failures, further confirms the good coupling achieved by applying flowable composite as a filled intermediate agent. Cohesive failures within the flowable resin were not recorded. This may be explained by the elasticity properties of the flowable intermediate, allowing for a stress-bearing ability higher than that of the hybrid resin composite.[29-30,34,36] The intimate adaptation of Gradia LoFlo to the resin substrate is in agreement with the satisfactory results of marginal/internal adaptation achieved on enamel and dentin, using flowable composite as an intermediate layer in Class I, II and V cavities.[37-39]

The quality of the repaired interface was also influenced by the temperature of the repairing composite. Gaps, detected when the cooled composite was used, are likely to behave as short- and/or long-term stress-raisers, probably leading to repair failure. On the contrary, the application of an uncured, pre-heated composite over a layer of previously cured flowable resin was the optimal combination in terms of repair strength and interfacial quality.

It is known that the photopolymerization of resin-based materials, relying on a complex free-radicals formation process and leading to a highly cross-linked network formation, may be influenced by the precuring temperature. An increased temperature may enhance the radicals mobility and polymerization rate, resulting in a superior crosslinking density and final degree of conversion of dimethacrylate-based monomers.[22,40-41] As a result, a warmed composite is prone to exhibit greater polymerization shrinkage than a cooler material.[32-33,42] In this investigation, since the fabricated composite build-ups represented a favorable configuration factor (C-factor), with a low ratio between bonded and unbonded composite surfaces, resin shrinkage presumably did not play a major role in the recorded bond strengths.[19] Hence, these results are clinically related to situations of low C-factor composite repair, such as the esthetic veneering of a pre-existing composite restoration because of color mismatch or unsatisfactory anatomic form. Clinical conditions with higher C-factor, such as the repair of restoration margins, could represent a concern, and this topic will be addressed in future investigations. The authors are aware that the amount of repairing composite in the current experimental setup might be viewed as a potential limitation, since the amount of resin placed in common clinical situations is smaller. Nevertheless, in the majority of methods used for bond strength measurement, specimen preparation procedures inevitably differ from clinical procedures. Comparably, these methods allow for the production of standardized test specimens and assess the potential differences between the investigated bonding approaches.[25]

On the other hand, bonding procedures performed on dental substrates, composite repair requires complete isolation of the operative field by a rubber dam. It has been reported that, by using the rubber dam, the intra-oral temperature around the treatment area becomes close to room temperature.[43] In a clinical scenario, after composite pre-heating, a time delay exists between dispensing the material, its placement and shaping of the substrate and light-curing, possibly decreasing resin temperature.[22,42] Thus, the detrimental effect of polymerization shrinkage expected at 37°C could also be minimized in high C-factor clinical conditions by this gradual cooling process.[40-41] However, even if pre-heated composite cools to 23°C before light-activation, benefits, in terms of interfacial strength and quality, are still present.

In this study, the effect of the pre-curing temperature was investigated only for a single brand of composite. As temperature may differentially affect the viscosity, depending on the resin composition (filler content, resin monomers), additional research should be done to determine if these results can be confirmed for other commercial materials.[22,24,28] Recently, a chairside preheating device that allows the operator to warm resin composites at 50°C-60°C immediately before placement into a tooth cavity has been marketed.[44] The effect of the additional heating on composite-to-composite strength and micromorphology should be further investigated.

Also, the clinician may not be aware of the type of resin composite needing re-intervention (especially when dealing with an old restoration performed by a different operator), and he or she may be faced with the question of materials compatibility. In this study, the composite substrate/repair and the flowable resin were based on UDMA monomers, while the bonding agent was based on Bis-GMA monomers. Although previous investigations have shown that a similar chemical nature of the bonded materials is not a prerequisite for superior interfacial coupling,[45-46] the *in vitro* performance of homogeneous (similar composition) and heterogeneous (dissimilar composition) repairs mediated by bonding and flowable resins, based on different monomers, deserves major attention in future research.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

1. The use of cooled (4°C) microfilled hybrid composite (Gradia Direct Anterior) as a repairing material resulted in poor composite-to-composite interfacial adaptation and lower repair strengths. The difficult adaptation of a highly viscous material to the roughened or intermediate agent-covered substrate probably explains these results.
2. Pre-heating of the repairing material to 37°C significantly enhanced repair strengths when no intermediate agent was used. Lower viscosity, achieved by heating the composite, may also justify the uniform composite-to-composite interfacial adaptation observed.
3. The use of a low-viscosity flowable composite (Gradia LoFlo) as an intermediate agent resulted in a significant improvement in repair strength under each of the three temperature conditions of the repairing resin.

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