



ORIGINAL ARTICLE

Repair bond strength of dual-cured resin composite core buildup materials



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ABSTRACT

The reparability of dual-cured resin composite core buildup materials using a light-cured one following one week or three months storage, prior to repair was evaluated. Two different dual-cured resin composites; Cosmecore™ DC automix and Clearfil™ DC automix core buildup materials and a light-cured nanofilled resin composite; Filtek™ Z350 XT were used. Substrate specimens were prepared ($n = 12$ /each substrate material) and stored in artificial saliva at 37 °C either for one week or three months. Afterward, all specimens were ground flat, etched using Scotchbond™ phosphoric acid etchant and received Single Bond Universal adhesive system according to the manufacturers' instructions. The light-cured nanofilled resin composite (Filtek™ Z350 XT) was used as a repair material buildup. To determine the cohesive strength of each solid substrate material, additional specimens from each core material ($n = 12$) were prepared and stored for the same periods. Five sticks ($0.8 \pm 0.01 \text{ mm}^2$) were obtained from each specimen (30 sticks/group) for microtensile bond strength (μTBS) testing. Modes of failure were also determined. Two-way ANOVA revealed a significant effect for the core materials but not for the storage periods or their interaction. After one week, dual-cured resin composite core buildup materials (Cosmecore™ DC and Clearfil™ DC) achieved significantly higher repair μTBS than the light-cured nanofilled resin composite (Filtek™ Z350 XT). However, Clearfil™ DC revealed the highest value, then Cosmecore™ DC and Filtek™ Z350 XT, following storage for 3-month. Repair strength values recovered 64–86% of the cohesive strengths of solid substrate materials. The predominant mode of failure was the mixed type. Dual-cured resin composite core buildup materials revealed acceptable repair bond strength values even after 3-month storage.

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Introduction

Core buildup restorations are often required for rebuilding severely damaged teeth with compromised resistance and retention prior to receiving indirect restorations. The improved strength, load transfer characteristics and durability along with advances in adhesive technologies directed conservative dentistry toward the use of resin composites as core buildup materials [1]. Resin based core buildup materials are available

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in self-cured, light-cured and dual-cured formulations. For the building of extensively damaged teeth, some clinicians currently prefer the use of dual-cured resin composites [2]. This type of resin composite is utilized to overcome the limitations of both extended chairside time [3], and depth of cure problems [4] that can occur with incremental layering techniques [5]. Dual-cured resin composite buildup restoratives combine the advantages of light- and self-cured mechanisms, in regard to a redox initiator system and photoinitiators [6]. Polymerization is mainly initiated by light activation in the superficial layers of the materials and by chemical activation in the deeper layers even when the curing light is severely attenuated [7].

During the treatment phase of full mouth rehabilitation some cases needs temporary cemented tentative restoration over the core buildup for a period of time until other steps of the treatment plan is achieved and the final indirect restoration is finally cemented. In some instances, such temporization could be debonded and part of the core material with or without the tooth chipped or partially fractured due to sudden biting on hard object before the tentative restoration is re-cemented. In this case, the clinicians are faced with the dilemma of selecting the optimal method for reconstruction [8]. Total replacement of defective core buildup materials results in a more invasive treatment with increased risk of complications and successive tooth loss in the future [9]. Additionally, core buildup replacement increases the cost of the procedure especially when a large portion of the restoration is clinically and radiographically intact [8]. At variance, repair provides an extended service and longevity for the existing restoration. The ability to repair light-cured resin composite materials was validated by many researchers [10].

In a literature survey, there were no data available on the repair potential of the dual-cured resin composite core buildup materials as to whether the fracture occurred shortly after preparation or later. Therefore, this study was carried out to evaluate the repair bond strength of stored (one week or three months) dual-cured resin composite core materials. The tested null hypotheses were (1) there is no difference among core materials repair strength values; (2) there is no difference in repair strength values with both storage periods prior to repair (one week and three months).

Experimental

Preparation of the substrate specimens

A total of 36 substrate resin composite specimens were prepared for this study. The materials, manufacturers, composition and batch numbers are listed in Table 1. The specimens were divided according to the core resin composite restorative material into three groups ($n = 12/\text{group}$). The first group included a light-cured resin composite restorative material [Filtek™ Z350 XT Universal Restorative, 3M ESPE, St. Paul, MN, USA, dentin shade (A_2)] and the other two groups included different dual-cured resin composite core materials; [Cosmecore™ DC core automix (Cosmedent America, Chicago, USA), dentin shade (A_2)]; [Clearfil™ DC core automix (Kuraray Noritake, Tokyo, Japan), Dentin shade (A_2)]. Each resin composite core material was inserted in a split Teflon mold (4 mm diameter \times 4 mm thickness) placed on top of a Mylar strip (Dental Technologies, Illinois, USA)

and a glass slab. The light-cured resin composite (Filtek™ Z350 XT) was applied in two increments of 2 mm each, while the dual-cured resin composite core materials (Cosmecore™ DC and Clearfil™ DC) were automixed before their application into the mold according to their manufacturers' instructions. The top of the increment was also covered with a Mylar strip and compressed with a glass slide to obtain a flat surface of the specimen. The top and bottom surfaces of the resin composite were cured from both sides for 20 s each using LED light curing unit (Blue Phase C5, Ivoclar Vivadent, Schaan, Liechtenstein) with an output light intensity of 450 mW/cm², periodically checked using an LED radiometer (Kerr Dental Specialties, West Collins Orange, CA, USA). After curing, the specimens were removed from the mold and checked using a magnification loupe (HEINE Optotechnik, Herrsching, Germany). The remaining fine flashes were carefully removed using a sharp lancet (Wuxi Xinda Ltd., Shanghai, China). Flashes were manually removed using a 220 grit SiC paper [11]. The base of each specimen was marked using an indelible type of markers (Sharpie®, Illinois, USA) of different colors to facilitate differentiation of the specimens. Specimens were then stored in artificial saliva [12] for one week or three months at 37 °C in a thermal incubator (Egyptian Medical Co., Cairo, Egypt). Artificial saliva solution was replaced weekly [13].

Repair of the substrates specimens

After the assigned storage periods, specimens were surface treated in two steps. First, the surface was wet-ground flat using a diamond wheel stone (Komet, Gebr.GmbH@ Co., Germany) [14]. Each specimen was then washed with tap water for 30 s and blotted dry. A digital caliper, (Mitutoyo digital caliper, Mitutoyo Corp., Kawasaki, Japan) was used to check that only 150–200 μm was removed from the height of each specimen. All specimens received acid etching with 37% phosphoric acid (Scotchbond etchant gel, 3MESPE, St. Paul, MN, USA) for 15 s followed by rinsing with water for another 15 s and then were air-dried for five seconds from a distance of 1 cm. Single Bond Universal Adhesive system (3M ESPE, St. Paul, MN, USA), was applied to the substrate surfaces using a microbrush (Shanghai Dochem Industries Co., Ltd., Shanghai, China) and gently agitated for 20 s. The adhesive was gently air dried for five seconds and light-cured for 10 s with light curing unit according to the manufacturers' instructions.

The treated substrate specimen was then inserted into another specially constructed repair mold (4 mm diameter \times 7.5 mm thickness) while the treated surface was directed upwards. Such height was obtained by assembling three split Teflon molds over each other; the first one with a height of 3.5 mm, the second one with a height of 2 mm and the last one with a height of 2 mm. Specimens were repaired using light-cured resin composite (Filtek™ Z350 XT) (shade B₂). A different shade was chosen for the repairing composite to enable visual identification and orientation of the repair interface during microtensile bond strength (μTBS) testing and failure mode observation [15]. The repairing composite was packed against the treated side of the substrate specimen incrementally (1.5 mm thick followed by 2 mm thick). Each increment was cured for 20 s. In order to test the cohesive strength of the tested materials, additional specimens of

Table 1 Material name/description, manufacturer, composition and batch number (#).

Material name/description	Manufacturer	Composition	Batch #
Filtek™ Z350XT (Shades A2, B2) Universal Restorative A visible light-activated nanohybrid methacrylate based resin composite	3M ESPE, St. Paul, MN, USA	Organic resin: Bis-GMA/UDMA/TEGDMA, and Bis-EMA Fillers: Combination of; 20 nm silica fillers (non-agglomerated/non-aggregated), 4–11 nm zirconia fillers (non-agglomerated/non-aggregated), and aggregated zirconia/silica nanocluster comprised of 20 nm silica and 4–11 nm zirconia particles Filler loading (78.5 wt%), 58–60 vol%	7018A2D 7018B2D
Cosmecore™ DC core automix A dual-cured core buildup hybrid methacrylate based resin composite	Cosmedent, Chicago, America	Matrix: Bis-GMA/UEDMA/Diacrylate Fillers: Ba-glass, silica 0.4–5 µm Filler loading: (70 wt%) 49 vol%	622-1A2
Clearfil™ DC, core automix A dual-cured core buildup hybrid methacrylate based resin composite	Kuraray, Tokyo, Japan	Resin: Bis-GMA/TEGDMA Filler: Silanated glass, silica Filler loading: (74 wt%) 52 vol% The particle size of inorganic fillers ranges from 0.04 µm to 23 µm Camphorquinone, Benzoylperoxide	#2942EU
Scotchbond™ Etchant gel	3M ESPE, St. Paul, MN, USA	35% by weight Phosphoric acid, 60% water and 5% Synthetic amorphous silica as thickening agent	N105148
Single Bond Universal Adhesive A single step one component self-etch adhesive system	3M ESPE, St. Paul, MN, USA	MDP Phosphate Monomer, Dimethacrylate resins, HEMA Vitrebond™ copolymer, filler, ethanol, Water, initiators and silane	41282

Bis-GMA: bisphenol A glycol dimethacrylate, Bis-EMA: bisphenol A ethyl dimethacrylate, DC: Dual cured, HEMA: hydroxyethyl methacrylate, MDP: methacry-loxydecyl dihydrogen phosphate, TEGDMA: Triethylene glycol dimethacrylate, UDMA: Urethane dimethacrylate, UEDMA: Urethane ethyl dimethacrylate.

4 mm diameter and 7.5 mm height were prepared from each resin composite core buildup material ($n = 12$) and stored in artificial saliva at 37 °C for one week and three months.

Microtensile bond strength testing

All specimens were fixed to the cutting machine (Isomet, low-speed saw, Lake Bluff, IL, USA) and serially sectioned to obtain multiple beam-shaped sticks. From each specimen, five sticks were tested, resulting in testing 30 sticks for each cohesive and repair group. The cross-sectional area ($0.8 \pm 0.01 \text{ mm}^2$) was confirmed with a digital caliper (Mitutoyo digital caliper, Mitutoyo Corp, Kawasaki, Japan). For μTBS testing, each stick was fixed to the modified ACTA μTBS jig [16] attached to a universal testing machine (Lloyd LRX, Lloyd Instruments Ltd., Fareham Hants, UK) using cyanoacrylate adhesive (Rocket, Dental Ventures of America Inc, Corona, CA, USA). The sticks were stressed in tension at a cross-head speed of 0.5 mm/min until failure. The load at failure was recorded in N, and the bond strength was calculated in MPa by dividing the load at failure by the cross-sectional area at the bonded interface.

Statistical analysis

The mean and standard deviation of each group were calculated. Comparison between repair groups was performed using Two-way Analysis of Variance (ANOVA) with repeated measures to test the significant effect of the core materials and the storage periods, as independent variable, as well as their interaction. One-way ANOVA was used to test the significant difference among the cohesive strength and the repair bond strength values of the different resin composite substrates at each storage period. A Bonferroni multiple-comparison post hoc test was used when indicated. The t -test was used to compare repair bond strength values of both storage periods for each resin composite core material. $P < 0.05$ was considered statistically significant. Data were analyzed using SPSS for Windows (Statistical Package for Social Sciences, release 15 for MS Windows, 2006, SPSS Inc, Chicago, IL, USA).

Mode of failure analysis

Both fractured sections of each stick (substrate side and repair resin composite side) were mounted on an aluminum stub, gold sputter coated and observed with a scanning electron microscope (SEM) (Scanning electron microscope 515; Philips, Eindhoven, Netherlands) at 100 \times magnification. Modes of failure were classified according to their main characteristics into 3 types; Type 1: Adhesive failure at the substrate side; Type 2: Mixed failure (adhesive at substrate side and cohesive in the adhesive layer) and Type 3: Mixed failure (adhesive at substrate side, cohesive in the adhesive layer and cohesive in the repair material). The frequency of each mode of failure was expressed as a percentage value.

Results

Two-way ANOVA with repeated measures (Table 2) revealed a significant effect for the core materials ($P < 0.001$), but not

for the storage periods ($P = 0.867$) or for their interactions ($P = 0.293$). Means, standard deviations and statistical significance of the repair and cohesive strength values (MPa) of all groups are represented in Table 3.

After one week and three months of storage, one-way ANOVA revealed a statistical significant difference among the repair bond strength values as well as the cohesive strength values of the tested core materials. Using Bonferroni multiple-comparison post hoc test, after one week, the repair μTBS values of the dual-cured resin composite core materials (Cosmecore™ DC and Clearfil™ DC) were not significantly different from each other but higher than the light-cured resin composite (Filtek™ Z350 XT). Following, 3-month storage, Clearfil™ DC value had the highest value followed by Cosmecore™ DC and Filtek™ Z350 XT as shown in Table 3. The t -test recorded no significant difference between the one-week and three-month cohesive strength and repair bond strength values of each resin composite core material (Table 3).

Mixed mode of failure [Type 3: Mixed failure (adhesive at substrate side, cohesive in the adhesive layer and cohesive in the repair material)] was the most common type of failure among the repair groups. Failure mode percentages of the tested repair groups are represented in Fig. 1. Representative scanning electron micrographs (SEM) for the most frequently detected failure modes are shown in Fig. 2.

Discussion

The findings of the present study revealed a significant difference among repair bond strength values of different resin composite core materials leading to the rejection of the first null hypothesis. So far, there is no published information on the reparability of the dual-cured resin composite core materials. The dual-cured resin composite core buildup materials revealed equal or higher repair bond strength values than those of light-cured one even after storage for three months prior to repair. This means that this material type has the potential to be successfully repaired. Despite using artificial saliva as an immersion solution to approximate the clinical situation; in the current study, none of the repaired group showed superior bond strength compared to the corresponding cohesive group with each storage time. Nevertheless, the repair strength of dual-cured core materials recovered 64–86% of their corresponding cohesive strength values while the light-cured material recorded a range between 76% and 81% of its corresponding cohesive strength values which was consistent with previous results for light-cured materials [11,17–19].

The two dual-cured resin composite core materials (Clearfil™ DC and Cosmecore™ DC) repair bond strength values were comparable, whereas the light-cured resin composite (Filtek™ Z350 XT) showed the lowest value following storage in artificial saliva at 37 °C for one week. Based on Brosh et al. [20], the binding between the old and the new composite in a repair case may occur by any of the following mechanisms; a chemical bonding with the organic matrix; a chemical bonding with the exposed filler particles, or micromechanical retention with the treated surface. Therefore, some possible explanations could be suggested. The first one is related to the amount of remaining active free radicals which are available in the substrate material and react with resin composite monomers, considered as a direct determinant for successful repair bond

Table 2 Results of two-way analysis of variance (ANOVA) with repeated measures for the repair groups.

Variable	Sum of Squares	DF	Mean Square	F-value	P-value
Substrate material	4450.74	2	2225.37	13.15	< 0.001
Storage period	4.86	1	4.86	0.03	0.867
Interaction	437.55	2	218.78	1.29	0.293
Error	4062.23	24	169.26		

n = 30/group.

Table 3 Microtensile bond strength (μ TBS) values [mean (standard deviation, SD)] in MPa of the tested groups.

	Repair μ TBS				Cohesive μ TBS			
	Filtek™ Z350 XT	Cosmecore™ DC	Clearfil™ DC	P-value	Filtek™ Z350 XT	Cosmecore™ DC	Clearfil™ DC	P-value
1-week	36.7(6.3)A	46.9(3.6)B	51.8(6.0)B	0.001	48(8.1)A	72.8(9.0)B	60.2(8.9)B	0.008
3-month	40.7(8.0)A	43.8(1.7)A	49.9(5.6)B	0.039	50.5(9.1)A	62.6(9.4)B	61.3(8.5)B	0.01
P-value	0.358	0.078	0.597		0.683	0.149	0.848	

N = 30 sticks/group. Within rows, different capital letters indicate significant differences in the bond strength values between groups (*n* = 30/group) (ANOVA, *p* < 0.05). Within columns, no significant differences in the bond strength values between groups were found (ANOVA, *p* < 0.05).

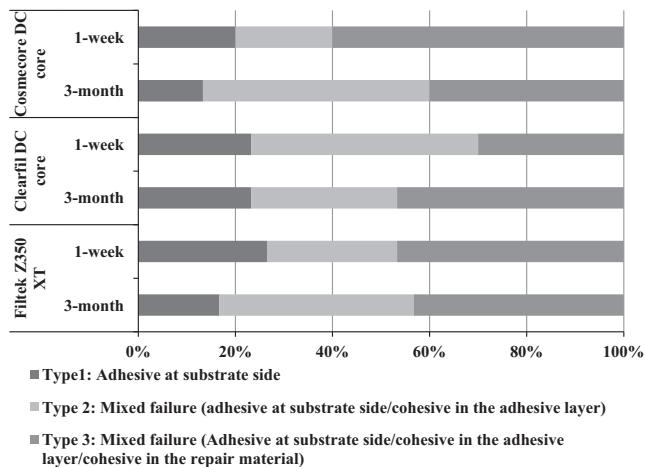


Fig. 1 Failure mode percentages of the tested repair groups.

strength [21,22]. Dall’Oca et al. [21], found that the remaining free active radicals were available up to 14 days after polymerization even in the absence of the oxygen inhibited layer. Kournetas et al. [5], showed higher amount of remaining double bonds (RDB) in the dual-cured resin composite core buildup material (Clearfil™ DC) compared to those found in the corresponding light-cured material (Clearfil Photo Core) of the same manufacturer. They referred this finding to the decrease in the camphorquinone content in the dual-cured resin.

Based upon this suggestion, we may be able to explain the relatively higher values recorded with Clearfil™ DC compared to those of Cosmecore™ DC dual-cured resin composite. Although both were composed of common resin matrix based on Bis-GMA/TEGDMA, Cosmecore DC contains additionally Urethane ethyl dimethacrylate (UEDMA) for partial substitution of BisGMA. UEDMA has been shown to improve C=C conversion [23–25], rendering it with less active double bonds compared to Clearfil™ DC. Even after three months

of storage in artificial saliva, the reparability of the dual-cured resin composite core materials (Clearfil™ DC and Cosmecore™ DC) demonstrated the same trend reported for one week results, where the lowest repair bond strength was reported for the light-cured resin composite (Filtek™ Z350 XT). Despite that the remaining free radical effect could be diminished after this storage period [21], the maintained repair bond strength could be referred to the availability of some degree of porosities which allowed better penetration of the intermediate adhesive agent particles. Also, these microporosities could be due to the plasticization and leaching of certain components out of the resin composite during storage [26,27].

Another reason for the recorded repair bond strength values is the reactivity of fillers of the tested resin composite after the proposed surface treatment [28]. The filler content in the light-cured resin composite (Filtek™ Z350 XT) is a combination of zirconia/silica, those of Cosmecore™ DC core are Barium-glass and silica, while for Clearfil™ DC core, fillers are silanated glass and silica. Loomans et al. [28], reported that materials which contain barium glass, silica (SiO₂) and pre-polymerized particles containing silica (clusters) showed high reactivity on surface treatment compared to zirconia fillers [29]. Eventually, zirconia fillers in Filtek™ Z350 XT seem to reduce the reactivity for surface treatments that might affect the reparability of this type of resin composite. Following the storage periods, substrate specimens in the present study received intermediate adhesive system containing silane agent. Silane has two main functional groups; where the silanol bonds to the silica of the composite filler, and the organofunctional group, co-polymerizes to the methacrylate of the bonding agent. It was reported that silane enhances the wetting, the infiltration of the adhesive system into the surface irregularities and the chemical coupling to the resin matrix and to exposed fillers [28] which could be an additional reason for the obtained repair bond strength values.

The storage period did not affect the repair strength, and thus, the second null hypothesis must be accepted. The lack of the effect of storage on the reparability was consistent with previous studies [17,30], although they used different materials

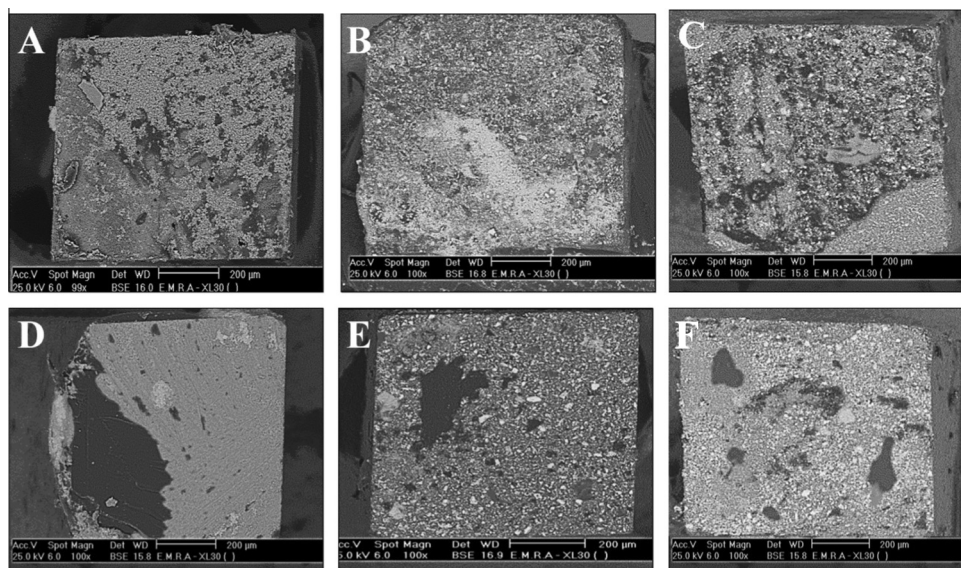


Fig. 2 Representative scanning electron micrographs (SEM) for the most frequently detected failure modes. Type 3 mixed failure mode (adhesive at substrate side/cohesive in the adhesive layer/cohesive in the repair material) of Filtek™ Z350 XT (A), Cosmecore™ DC (B) and Clearfil™ DC (C) core materials repaired after one week storage in artificial saliva. D, E and F represent Type 2 mixed failure mode (adhesive at substrate side/cohesive in the adhesive layer) recorded for Filtek™ Z350 XT, Cosmecore™ DC and Clearfil™ DC resin composite core materials repaired after three months of storage in artificial saliva.

and different storage periods. After three months of storage, no significant difference was found among the cohesive groups. This result also was in agreement with many other studies [13,17,30,31]. The development of resin composite allowed it to be more resistant to storage even under conditions that mimic the clinical situations. It worth mentioning that this study assumed that the fractured part was accessible and thus can be treated with light-cured resin composite. To check whether comparable repair bond strength could be obtained in case self- or dual-cured resin composite was used as repair material for inaccessible areas or light cure, further investigations are necessitated.

In general, the ideal core buildup materials should provide adequate stress distributions of forces reducing the probability of tensile and compressive failures, and provide high reparability. Dual-cured resin composites have proven to be able to successfully take a part in the preferred materials used for this purpose.

Conclusions

Dual-cured resin composite core buildup materials revealed acceptable repair bond strength values even after 3-month storage.

Conflict of Interest

The authors have declared no conflict of interest.

Compliance with Ethics Requirements

This article does not contain any studies with human or animal subjects. However, the study protocol was following the rules

of the local ethical committee of the Faculty of Oral and Dental Medicine.

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