Original Article

Effect of Different Formulations of Dentin Replacement Materials and Aging on the Flexural Strength of the Overlying Resin Composite

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Abstract

Introduction: This study aimed to evaluate the flexural strength (FS) of dentin replacement materials, including; fiber-reinforced composite, bulk-fill flowable composites, and resin-modified glass ionomer (RMGI), layered with nanohybrid composite (NH) at different storage times. **Methods:** A total of 100 specimens were prepared (n=10) and divided into five groups depending on the dentin replacement material used, and a control group with conventional NH incrementation. Each group was further subdivided into two groups according to the time of the FS testing; 24 hours or 6 months. The specimens were subjected to a 3-point bending test till failure. The comparison between the base materials and time was made using the two-way ANOVA, while the comparison between the base materials within each time interval was made using the one-way ANOVA and the Tukey's post hoc test. Additionally, the comparison between the immediate and aged FS within each group was made using the Student's t-test. **Results:** After 24 hours, the resin-based, bulk-fill dentin substitutes layered with NH and the incrementally placed NH, showed a higher FS than the RMGI. However, after 6 months, all groups showed a significant decrease in FS, with the exception of the RMGI group, which showed a significant increase. **Conclusion:** Resin-based dentin replacements showed better or similar reinforcement effects compared to conventional composite incrementation, when tested immediately or after 6 months. Aging over 6 months had a deteriorating effect on the FS of all composite resin materials, while it improved the FS of the overlying composite resin in the RMGI group

Keywords: Flexural Strength, Composite Resins, Dental Materials, Fiber-Reinforced Composite.

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Introduction

Composite resin has been the restorative material of choice for years, and recent advances in materials and adhesive systems have expanded the scope of indications. However, composite resin has some drawbacks, such as polymerization shrinkage, which induces stresses at the tooth and restoration interface, leading to impairment of marginal integrity, difficulties in application technique (1,2), and possible fracture of the restoration due to a difference in the physical and mechanical properties between the tooth and restoration (3) To overcome such drawbacks, the use of the base material in a sandwich/laminate technique has been proposed (4).

The use of a two-layer restorative approach corresponds to the modern biomimetic concept of restorative dentistry. Biomimetics is the study of the structure and function of tooth tissue as a model for the design and manufacturing of materials and techniques for restoring or replacing teeth. This includes the replacement of missing dental hard tissues with restorative materials that mimic natural tissue in terms of their mechanical

properties (5). Thus, the use of dentin replacement material of a different formulation with a nanohybrid composite resin capping layer can meet such an emerging concept. Dentin replacement offers several advantages to the restorative procedure, such as improving the adaptation of the restoration to deep gingival seats in composite cavities due to their reduced viscosity, thereby facilitating the restoration procedure, which would consequently be less time consuming (5,6).

Over the years, several dentin replacements, formerly known as base materials, have been developed for the use of the laminate technique, also known as the sandwich technique (6). In this technique, a base material (i.e. dentin replacement material) is placed under the restorative material of choice. The laminate/sandwich technique can be "open", in which the base material is exposed to the oral environment as in class II restorations, or "closed", when the overlying restorative material is placed along the cavity margins so that the base material is not exposed to the oral environment (7,8).

The resin-modified glass ionomer (RMGI) is the standard material for the layered technique due to its chemical adhesion to the tooth and ease of manipulation. In the course of the development of composite resins, bulk-fill materials were introduced that can be cured in 4 mm thick increments, which were specially developed for dentin replacement. These materials contain polymerization modulators to reduce the polymerization shrinkage stress at interface of the tooth restoration. The further development of bulk-fill dentin replacements has led to the randomly oriented E-glass fibers being incorporated into a resin matrix, creating a so called "semi-penetrating polymer network" that enables stress to be transferred from the polymer matrix to the fibers (9). Tanner *et al.* have considered the use of a two-layer fiber reinforced composite resin as a base material in combination with particulate-filled composite as the top layer, which represents a biomimetic approach as it structurally mimics dentin and enamel under load (10). In general, manufacturers recommend all bulk-fill dentin replacements to be capped with a layer of universal nanohybrid restorative material in order to ensure durable restorations (10, 11).

The annual failure rates of composite resin restorations have not changed significantly over the past two decades. However, the cause of failure has shifted from a primarily biological one with secondary caries and postoperative hypersensitivity to a more mechanical cause such as tooth or restoration fractures (12), emphasizing the mechanical evaluation of materials while simulating complex oral conditions in dental research. Therefore, this study was conducted to evaluate the flexural strength of various dentin replacement formulations layered with a nanohybrid composite resin and the combined structure as used in clinical situations. The null hypotheses were that, first, the use of different dentin replacement formulations did not affect the flexural strength of the two-layer structure. Second, storage time did not affect the flexural strength of the combined structure.

Materials and Methods

1. Study Materials

Four composite resins and one resin-reinforced glass ionomer material were used in this study. Materials, description, compositions and their lot numbers are summarized in Table I.

Table I. Materials (and manufacturer), description, compositions and lot numbers.

Material & Manufacturer	Description	Composition	Lot Number
Filtek Z250 XT, (USA, St. Paul, 3M ESPE.)	Nano-Hybrid Universal Restorative, light-activated composite resin material	Fillers: Surface-modified zirconia/silica with a median particle size of approximately 3 microns or less. Non-agglomerated/non-aggregated 20 nm surface-modified silica particles. Filler loading of 82% by wt (68% by vol). The resin system: Bis-GMA*[1], UDMA*[2], Bis-EMA*[3], PEGDMA*[4] and TEDGMA*[5]	N797176
SDR® (Smart Dentin Replacement), USA, Caulk, DENTSPLY.	Bulk fill, flowable composite resin restorative material	SDR™ patented urethane dimethacrylate resin, di-methacrylate resin, di-functinal diluent, barium and strontium alumina-fluoro-silicate glasses (68% by wt, 45% by vol), photoinitiating system and colorant.	170711
X-tra base, Germany, VOCO GmbH.	Bulk fill flowable, self-leveling base or liner composite.	Aliphaticdimethacrylate, Aromaticdimethacrylate (Bis-Ema), inorganic filler, fumed silica (75% by wt, 60.2%by vol)	1708155
Ever-X Posterior, Japan, Tokyo, GC Corporation.	Bulk fill, Light cured, fibre-reinforced composite for dentin replacement.	Bis-GMA, PMMA*[6], TEDGMA, Short E-glass fiber filler, Barium glass 74.2 wt%, 53.6 vol%	1704181
Fuji II LC, Japan, Tokyo, GC Corporation.	Light cured, Resin reinforced glass-ionomer restorative	Liquid: Poly acrylic acid Powder: Al2O3-SiO2-CaF2 glass and HEMA*[7] urethane dimethacrylate.	1708173

*[1]Bis-GMA: Bisphenol A-Glycidyl Methacrylate. *[2]UDMA: Urethane Dimethacrylate. *[3]Bis-EMA: Ethoxylated Bisphenol A glycol Dimethacrylate. *[4]PEGDMA: polyethylene glycol dimethacrylate*[5]TEDGMA: Tetraethyleneglycol Dimethacrylate. *[6]PMMA: polymethyl methacrylate. *[7]HEMA: Hydroxyethyl Methacrylate

2. Procedures

Mold Fabrication

Two silicone molds were made from addition polyvinyl siloxane. This was done by making composite resin bars of the required dimensions. The bars were fixed in a plastic container with a diameter of 3 cm using cyanoacrylate. The putty and light consistencies of the addition silicone were used to capture the bar dimension, creating a negative replica of the bar.

The final specimen dimensions in this study were chosen to be 12 mm (length) x 2 mm (width) x 6 mm (depth). Therefore, two molds with depths of 4 and 6 mm were made. The 12x2x4 mm mold was used to apply the base/dentin replacement material, while the other (12x2x6 mm) was used to create a 2 mm space above the base material for the nanohybrid composite resin capping layer.

Test Specimen Fabrication

A total of 100 specimens were fabricated (n=10) and divided into 5 groups depending on the base material used. Each of the five groups was subdivided into two groups according to the time of the flexural strength test, namely 24 hours and 6 months. The depth was chosen to simulate clinical situations with deep gingival seats in class II cavities. In order to achieve such dimensions, the

bulk-fill base material was applied in a single increment of 4mm into the first mold. The excess material was removed before curing and a Teflon tape was placed over the material, followed by a glass slab to obtain a smooth and flat surface. The glass slab and Teflon tape were then removed and the material was cured using an LED curing device (Radii plus LED curing light, SDI. Light output: 1500 mW/cm²) according to the manufacturer's instructions. The recommended curing time for Group 1 (X-tra base, XB) was 10 seconds, while it was 20 seconds for Group 2 (SDR), Group 3 (RMGI), and Group 4 (EverX posterior, EX). Three Overlapping curing exposures were used. The light-curing tip was brought as close as possible to the surface of the material and the tip was directed perpendicular to the surface. The light intensity was frequently checked using a radiometer (Demetron, LED Radiometer, Kerr) for each test group.

For the RMGI, two capsules were used for each specimen. The capsules were activated and placed in an amalgamator (Ultramat 2, SDI) and mixed for 10 seconds according to the manufacturer's instructions. During the RMGI application, two consecutive 2 mm thick layers were injected into the mold and cured, resulting in a 4 mm thick increment. Next, the specimens were removed from the first mold and transferred to the deeper mold (12x2x6 mm) which was used to apply a top layer of the nanohybrid composite resin packed using a ball burnisher, and the excess was removed before curing. Figure 1 shows an illustration of the final test specimen layers.

Control specimen Fabrication

The control group (Group 5, NH) consisted of specimens with nanohybrid composite resin incrementation and without base material. The specimens were prepared by incrementally applying a 2 mm nanohybrid composite resin. Three increments were applied in succession to make a specimen of 6 mm deep. A graduated periodontal probe was used to ensure that the increments are no thicker than 2 mm. The nanohybrid composite resin was cured for 20 seconds according to the manufacturer's instructions. Half of the specimens were stored in an incubator at 37°C for 24 hours (Arthermo, Italy) and the flexural strength was then assessed. Figure 2 shows an illustration of the increments applied in the control group of the nanohybrid composite resin.

Artificial Aging Procedure

The other half of the specimens in each group were stored in distilled water in glass vials at 37°C in an incubator for 6 months. The distilled water was replaced weekly. Then the specimens were subjected to a flexural strength test.

Flexural Strength Test

Each specimen was placed in a specially fabricated metallic attachment with a 2.5 mm engraved vertical slit to secure the specimen during testing. The metallic attachment was secured to the lower jaw of the machine, and a bi-beveled steel chisel was secured to the upper jaw of the machine. A three-point bending test was conducted to measure flexural strength on a computerized universal testing machine (INSTRON, Illinois Tool Works Inc. USA) using a load cell unit of 5 kN at a crosshead speed of 1 mm/min. The data was recorded using the Bluehill Lit software. The flexural strength was measured in megapascal (MPa).

Statistical Analysis

This study was carried out to examine the impact of using different base materials layered with nanohybrid composite resin on the flexural strength after 24 hours and 6 months. The comparison between the base materials and time was made using the two-way ANOVA, while the comparison of the materials within each time interval was made using the one-way ANOVA. In addition, a Tukey's post hoc test was used in the case where the one-way ANOVA showed statistical significance. The comparison between the immediate and aged flexural strength for each group was made using the Student's t-test.

Results

The two-way ANOVA showed that the base materials influenced the flexural strength with an F-value of 109.8 and $P \leq 0.0001$. In addition, aging had also an effect on the flexural strength with an F-value of 438.1 and $P \leq 0.0001$. The interaction between the two variables had an effect on the flexural strength with an F-value of 75.01 and $P \leq 0.0001$.

The data for the 24 hours groups were analyzed using the one-way ANOVA and the Tukey's post hoc test. The materials and their flexural strength in MPa (mean ± SD) are given in Table II in descending order of the mean flexural strength, i.e. SDR (128.5 ± 16.03), XB (125.2 ± 12.7), NH (108.5 ± 12.09), EX (95.66 ± 14.87), and RMGI (21.62 ± 6.84). Similarly, the data for the aged groups (6 months) were analyzed using the one-way ANOVA and the Tukey's post hoc test. The materials and their flexural strength in MPa are also given in Table II in descending order of their mean, i.e. EX (72.55 ± 11.21), SDR (60.72 ± 12.66), XB (46.13 ± 6.287), RMGI (39.72 ± 7.105), and NH (35.11 ± 10.58).

Table II. Mean ± SD of flexural strength in MPa of the materials tested at 24 hours and 6 months.

Material	Flexural Strength after 24 hours in MPa (Mean ± SD)	Flexural Strength after 6 months in MPa (Mean ± SD)	P-Value	Percentage of change
Group 1: X-tra Base (XB)	125.2^{A} (±12.7)	46.13^{c} (±6.287)	0.0001**	-63.1%
Group 2: SDR (SDR)	128.5^{A} (±16.03)	60.72b (±12.66)	0.0001**	-52.7%
Group 3: RMGI (RMGI)	21.62^{C} (±6.84)	39.72^{c} (±7.105)	0.0001**	83.7%
Group 4: EverX Posterior (EX)	95.66^{B} (14.87)	72.55^{a} (±11.21)	0.0007**	-24.1%
Group 5: Composite Increment/Control Group (NH)	108^{B} (±12.09)	35.11^{c} (±10.58)	0.0001**	-67.4%

At P≤0.05 (*) statistically significant, (**) highly statistically significant
-Comparison between the flexural strength values of specimens tested after 24 hours and 6 months are presented within the same row.
-Comparison between different materials are presented within each column.
Superscripts with different letters are statistically significant at P≤0.05
Superscripts in upper case letters represent the comparison between different materials after 24 hours. Superscripts in lower case letters represent the comparison between different materials after 6 months.

With the same base material, the Student's t-test showed that aging has a significant effect on the flexural strength of the materials. After aging for 6 months, there was a significant increase in the flexural strength of RMGI by 83.7%. In addition, there was a significant decrease in the flexural strength of SDR, XB, EX, and NH by 52.7%, 63.1%, 24.1%, and 67.4%, respectively (remarked by "–" in Table II).

Discussion

When restoring the dentition, dentists usually use direct additive adhesive techniques, in which dental materials are inserted into the defects and are then light-polymerized. This conserves the tooth structure and the tooth integrity is thereby preserved (13). Flexural strength was employed in this study to evaluate the mechanical properties of dentin substitutes capped with nanohybrid composite resin as would be used clinically.

Within the limitations of this in vitro study, it was shown that different formulations of dentin substitutes and storage time have a significant impact on flexural strength. Thus, the first and second null hypotheses were rejected.

Specimen dimensions were selected according to the proposed mini-flexural strength test, which uses smaller and more clinically realistic specimens (13-15). It has been argued that the ISO specimen dimensions are challenging to fabricate without defects and require the use of multiple overlapping irradiations due to comparatively smaller light curing tips. Furthermore, these ISO specimens are not clinically relevant as the mesiodistal dimension of the molars are approximately 11 mm and the cervicoincisial dimensions of the central incisors rarely exceed 13 mm. In addition, the ISO specifications require more expensive materials (16) so the mini-flexural test has been adopted. The mini-flexural test uses specimens that are 12 mm in length instead of the 25 mm required by ISO specifications, making the specimen dimensions more clinically relevant and less time-consuming to fabricate (17).

The aim of this in vitro study was to simulate a deep cavity, where it is indicated and preferred to use dentin substitutes to facilitate the restorative procedure. Therefore, the depth of the specimen was adapted to the clinical application of the material, i.e. 4 mm of bulk-fill dentin substitute and a 2 mm nanohybrid composite capping layer. It has been shown that changes in the length or height of flexural strength test specimens do not affect the result of the test (16) A study by Calabrese *et al.* demonstrated the importance of layering composite resin specimens; as they would be used in clinical settings to correctly assess of the mechanical performance of such materials (18).

Flexural strengths obtained after storage in water at 37°C for 24 hours showed that XB and SDR had the highest flexural strengths compared to other groups. This may be due to the higher weight percent filler in both materials, which is 75 and 68 weight percent in XB and SDR, respectively. Kim et al. (19) and Tsujimoto et al. (20) found a positive correlation between filler loading and mechanical properties including flexural strength.

These results are in agreement with Öznurhan *et al.* (21) who found that XB and SDR had higher flexural strength than a nanohybrid composite resin. They justified their results with different filler systems and volumes in the materials. On the other hand, Garoushi *et al.* (22) reported higher flexural strengths for short fiber-reinforced composite and XB compared to SDR. The authors rejected the positive correlation between filler loading and mechanical performance and explained it for the short fiber-reinforced composite through the stress transfer from the fibers to the matrix due to the semi-interpenetrating network and a highly cross-linked matrix. The conflicting results in the literature are due to test methods, where evaluating a single layer of material in the above studies may produce different results than testing a two-layer structure that simulates the clinical situation with its complex variables.

The lower flexural strengths of NH were unexpected due to its high filler content of 82% by weight. The reason for such decreased flexural strength may be the sensitivity of the composite resin increment technique. Several disadvantages have been attributed to this technique, such as the incorporation of voids in the restoration body as a result of air entrapment between the increments and the increased risk of contamination between the composite layers (1). In addition, improper increments can result in areas with uncured composite resin. This leads to a reduced strength of composite resin restorations (23).

The RMGI group had the lowest flexural strength. This can be explained by the lack of reinforcing fillers, which makes it the weakest structure among the materials tested. This agrees with Alrahla (24), who compared the flexural strength of RMGI, SDR and a nanohybrid composite resin. Their results showed significantly higher flexural strengths for SDR and nanohybrid composite resin compared to RMGI.

However, the results changed as shown in Table II after immersion in distilled water for 6 months. The data showed a significant decrease in flexural strength for all groups except RMGI. This can be explained by the hydrolytic effect of water on the composite resins. Water degrades resin-based materials due to one or more of the following reasons. First, as polymer molecules are polarized, they attract water, which leads to the diffusion of water between the polymer chains. The presence of water between the chains leads to their separation, which in turn allows further penetration of water at a higher rate and in a larger amount. Therefore, plasticization occurs because polymer chains become less densely packed and can move freely within the structure, which softens the material. As a result, mechanical properties such as strength, fracture toughness and modulus decrease. Second, the presence of voids within the structure can allow water uptake and the breakdown of polymer chains (25). Third, water has an adverse effect on the sealant at the interface between the fillers and the matrix or the glass fiber and the matrix by causing rehydrolysis, which leads to a reduction in mechanical properties. (26) Another reason for the decrease in the flexural strength in this study can be the immersion of the specimen in water, which increases the surface area exposed to the aging medium and consequently increases its effectiveness.

The variations between the materials tested may be due to the different monomers that constitute the matrix. The matrix plays an important role in the longevity and durability of composite resins as it consumes around 20-40% of the structure (27). The matrix can have a synergistic effect with the fillers in increasing the mechanical properties of the material, or an antagonistic effect in degrading them in the long term, with possible loss of unreacted monomers (28).

The EX group showed a decrease in its flexural strength of 24.1%. However, this was the highest flexural strength after aging due to its matrix which containing Bis-GMA, PMMA and TEDGMA. Bis-GMA is a high molecular weight (513 g/mol) monomer of high viscosity. Although a high viscosity leads to limited mobility and lowered DC (29), due to its hydroxyl group it offers a strong intermolecular bond rather than intramolecular interaction and forms a rigid backbone (30). Moreover, due to its high molecular weight, BIS-GMA shows hydrophobicity. This was shown in a study by Alshali et al. (29) who reported a log-P of 5.53 for Bis-GMA, which means that this polymer is relatively hydrophobic. Log-P is a distribution coefficient method used to measure hydrophobicity. It gives an insight into the diffusivity of water into a dental resin. Thus a matrix containing Bis-GMA should be relatively stable in water. However, in order to improve the handling properties of the material and to enable the incorporation of a higher proportion of fillers, Bis-GMA must be diluted with a comonomer of lower viscosity (28).

In the case of EverX posterior, TEDGMA takes on this task. This was in agreement with Abdul-Monem et al.,

(31), who compared EverX posterior with a nanohybrid composite resin. The drop in flexural strength of EverX posterior was much less than that of the nanohybrid composite resin group.

The NH group showed a 67.4% reduction in flexural strength. This can be attributed to a decrease in DC, which could be due to the increase in distance from the light curing tip to the deepest increment of the resin (32). Ideally, the light-curing tip should be a maximum of 3 mm away from the composite increment. Increasing the distance between the light curing tip and the composite resin increment leads to a decrease in the light intensity that reaches the deepest layer, which in turn reduces the DC.

According to a review by Malhotra and Mala (33) every 1 mm greater distance from composite resin can lead to a 10% reduction in DC.

In clinical situations, the gingival seat of deep Class II preparations can reach a depth of 6.3 mm from the occlusal surface. This means that composite resin placed at this depth is likely to receive very low irradiation and will subsequently diminish the DC (34). Therefore, a reduction in the durability of the material and mechanical properties can be expected (35).

In a study evaluating light transmittance through bulk-fill materials and its relationship to mechanical properties, it was found that X-tra base experienced a greater decrease in light transmittance than SDR. This can explain why SDR has the second highest flexural strength after storage. The study concluded that the DC is a product-dependent property and should not be generalized to all bulk-fill materials (36).

The RMGI group was the only group that showed a significant increase in flexural strength of 83.7% after 6 months of storage. This unique phenomenon could be explained by the maturation of RMGI through post-cure reactions.

Conclusion

Within the limitations of this study, resin-based dentin replacements showed better or similar reinforcement effects compared to conventional composite resin increments when tested immediately or after 6 months. Aging over 6 months had a deteriorating effect on the flexural strength of all composite resin materials, while it improved the flexural strength of the overlying composite resin in the RMGI group. Therefore, it can be suggested that resin-based bulk-fill dentine replacement materials are an efficient alternative for restoration of deep cavities.

Conflict of Interest

The authors declare that there is no conflict of interest.

Acknowledgments

None

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