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Influence of Heliobond on Microtensile Bond Strength of a New **BIS-GMA Free Versus BIS-GMA Containing Composite Resin** Restoration

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Abstract

Edited by: Aleksandar Iliev Edited by: Aleksandar liev Citation: Elsebaey L, Abdellatef W, Alahmed B, Elmarakby AM. Influence of Heliobond on Microtensile Bond Strength of a New BIS-GMA Free Versus BIS-GMA Containing Composite Resin Restoration. Open Access Maced J Med Sci. 2023 Aug 10; 11(D):115-123. https://doi.org/10.3890/appine.2023 11206 https://doi.org/10.3889/oamjms.2023.11705 Keywords: Microtensile bond strength Free Bis-GMA resin composite (Admira) Bis-GMA cortaining resin composite (Admira): Bis-GMA cortaining resin composite (Grandio); Heliobond *Correspondence: Ahmed Mohamed Elmarakby, Department of Operative Dentistry, Faculty of Dental Medicine, Al-Azhar University, Assiut Branch, Assiut, Egypt. E-mail: drahmedmarakby@yahoo.com Received: 15-May-2023 Revised: 01-Jun-2023

Copyright: © 2023 Labib Elsebaey, Weam Abdellatef, Bader Alahmed, Ahmed Mohamed Elmarakby Funding: This research did not receive any financial support

Competing Interests: The authors have declared that no

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AIM: The main objective of this study was to assess the micro-tensile bond strength (uTBS) of a free bisphenol-adiglycidyl-ether-dimethacrylate (Bis-GMA) resin composite restorative material compared to a Bis-GMA-containing resin composite following the application of a hydrophobic coating (heliobond).

MATERIALS AND METHODS: A flat occlusal dentin surface was exposed in a total of eighty extracted teeth that were removed for periodontal reasons. Teeth were divided into two main equal groups according to the type of applied filling materials: BIS-GMA-free versus BIS-GMA-containing resin composite (n = 40). Each main group was subdivided into two equal subgroups (n = 20) according to the application of Heliobond (hydrophobic resin coating). Heliobond has been applied after adhesive application and before resin composite application. The first group was restored by a Free Bis-GMA Resin Composite (Admira, Voco, Germany); the second group was restored by a Bis-GMA-containing resin composite (Grandio, Voco, Germany). Each tested restorative material was applied and cured according to the manufacturer's instructions.

RESULTS: Regardless of different composite and adhesive types, there was a statistically significant difference (p < 0.05) among all subgroups. Specimens with Heliobond recorded a higher μ TBS mean value (30.46 ± 6.7 megapaskal [MPa]) than groups without Heliobond, which recorded μ TBS mean value (23.95 ± 9.02 MPa).

CONCLUSION: Application of an extra hydrophobic layer coating (Heliobond) has improved the performance of the µTBS of the adhesive systems utilized with the new BIS-GMA-free versus BIS-GMA-containing composite resin

Introduction

Adhesive restorations are frequently used as standard operating procedures in minimally invasive restorative dentistry. The bond strength test on enamel and dentin is used to assess the adhesive materials' ability to adhere. Tensile bond tests and shear bond tests have often been used for this purpose. The micro-tensile bond test is being used more often to evaluate more modern adhesive materials as a result of improvements in the bonding capability of the materials [1].

The primary objective of bond strength testing is generally acknowledged to be the comparative assessment of the adhesive capability of materials. Poor micro-tensile bond strength (µTBS) could lead to gap formation and subsequent micro-leakage at the tooth restoration interface. Micro-leakage had been thought to be an important factor in causing postoperative tooth sensitivity, marginal discoloration, and recurrent caries. This explains why micro-leakage examinations at the margins of restorations have been routinely done. Different cavity designs and filling methods have been assessed in the micro-leakage experiments. The contraction stress of composite resin during polymerization, which is larger than the bond strength, causes the restoration to separate from the cavity walls [1].

The most common restorations utilized nowadays are resin composite restorations that are held in place with an adhesive resin. They resemble dentin in terms of its physical and mechanical qualities. Numerous studies have been conducted to enhance the qualities of resin composite restorative materials since they were first introduced in 1960 [2].

Due to its higher cosmetic quality, ease of administration, and increased mechanical strength, resin composite based on bisphenol-a-diglycidylether-dimethacrylate (Bis-GMA) has become essential for dental restoration, although there are still issues.

Polymerization shrinkage in the monomer phase is a significant problem. post-operative sensitivity, marginal discoloration, secondary caries, cuspal displacement, and even cracks and fissures in healthy tooth structure that might result from polymerization shrinkage and subsequent contraction pressures [3].

Methacrylate-free resin composites have been developed to combat this issue in the hopes of improving biocompatibility and lowering shrinkage stress [4]. Recently, an organically altered ceramic known as Ormocer was introduced. It combines the toughness of glass with the characteristics of resin by using silicon dioxide as an inorganic basis and polymerizable organic chemicals as the organic components. The purpose of this substance is to enhance not only esthetics but also μ TBS, enabling a reduction in polymerization shrinkage and surface roughness as well as caries prevention. Additionally, because it is free of Bis-GMA and all other types of typical methacrylates, it is thought to be innocuous and improves biocompatibility without raising any questions about cytotoxicity [5].

Because contraction forces developed during the polymerization of dental restorative composites placed in a restricted setting cause tension in the material, with possible subsequent distortion of the bond to the tooth, it has been hypothesized that there is a highly significant correlation between polymerization shrinkage and µTBS [6]. Additionally, the incompatibility of the adhesive and the restorative material as well as the surface tensions of the two components in contact with one another may have an impact on the strength of the connection between the tooth and the restorative materials, i.e., the tooth-restoration interface [7]. The magnitude of this shrinkage affects the tension state created at the contact point of composite or dental structure and frequently jeopardizes the bond's integrity in this area. The geometric shape of the cavity also affects the polymerization shrinkage of composites. The tension caused by composite shrinkage may be greater than the bond strength to the cavity walls when the ratio of bounded to unbounded surfaces (C factor) is more than two, leading to marginal gaps [8]. Clinically, marginal leakage, poor anatomic shape, and proximal contacts arise when these issues are combined with improper placement technique and finishing errors, which ultimately reduce the restoration's durability and longevity [9], [10], [11].

It has been reported that short- and longterm resin-dentin bonding of universal adhesives can be improved by an additional hydrophobic resin coat [12], [13], [14], [15], [16]. The application of an additional hydrophobic resin coat aims to increase the thickness and uniformity of the adhesive layer and reduce fluid flow across the adhesive interface [12], [13], [14], [15], [17], [18]. This less permeable layer can help prevent the degradation of eroded dentin. Simplified adhesives, such as universal adhesives that combine hydrophilic and hydrophobic monomers in a unique bottle, promote the creation of an adhesive interface that lacks a non-solvated hydrophobic resin coating [19]. The formed hybrid layer is highly permeable to water from the oral environment and to water fluxes from dentinal tubules [19]. A more hydrophilic adhesive has a higher water sorption rate, resulting in fast hydrolytic degradation of the hybrid layer [19], [20], [21], [22]. Several researchers have advocated the use of an additional hydrophobic resin coat to improve the bonding performance of adhesives [16], [23], [24], [25].

The main objective of this study was to assess the Influence of Heliobond on μ TBS of a free Bis-GMA resin composite restorative material compared to a Bis-GMA-containing resin composite following the application of a hydrophobic coating (HC).

Materials and Methods

Non-carious eighty extracted human sound molars were collected from patients in educational clinics of the Faculty of Dental Medicine, Nahda University. The patient's teeth suffered from grade III mobility for periodontal reasons, so they required extraction. Teeth were collected after approval from the local ethics committee (#06\11\22 NUB-MREC). They were disinfected with 0.5% chloramine and stored in distilled water until use.

Specimens' preparation

The roots of each tooth were embedded in acrylic resin blocks vertically along their long axis apically from the cemento-enamel junction using Hollow metallic cylindrical templates (30 mm in diameter and 25 mm in height). The occlusal third of the crown was removed from all teeth using a diamond saw in a cutter machine with water cooling (Isomet, Buehler, Lake Bluff, IL, USA) to obtain a flat dentin surface. To confirm the absence of enamel on the dentin surface, careful examination was performed under a stereomicroscope (Olympus SZ40, Tokyo, Japan) at 30× magnification. The exposed dentin surfaces were polished with wet #600-grit silicon carbide abrasive paper (SiC) for 30 s to standardize the smear layer [11]. Teeth were cleaned using a manual scaler to remove all calculus and remaining soft tissues. All steps for specimen preparation are discussed in Figures 1a-e, using the article of Russo et al. as guidance [26], [27].

Sample size calculation

The sample size calculation was performed online (www.sealedenvelope.com), accessed on



Figure 1: Specimen preparation. (a) Each tooth was encased in acrylic resin inside a steel mold. A square section metal pin (asterisk) was inserted at the bottom end of the acrylic resin block to facilitate the positioning of samples onto the precision sectioning saw. (b) Each tooth was sectioned perpendicularly to its long axis, a 1st time in order to remove the occlusal enamel, and a 2^{nd} time, to obtain a 2 mm-thick slab of mid-coronal dentin framed by acrylic resin. (c) Metal device for specimen construction. Each dentin slab, framed by acrylic resin (asterisk), was placed between the two metal plates (full arrow) and the silicon sheets (blank arrow), which were then joined together with four screws. (d) Assembled custom made device. (e) Nine perfectly aligned conical frustum shaped build-ups, whose smaller base was bonded to the dentin surface, were constructed on both surfaces of each dentin slab Russo et al. [27]

February 24, 2023. The sample size was determined using the uTBS mean ± standard deviation values for Universal adhesive on sound dentin reported in the literature (49.8 ± 5.3 megapaskal [MPa]) [27], [28], [29]. To detect a difference of 8 MPa between the tested groups at a significance level of 5%, with a power of 80%, and using a two-sided test, the minimum sample size was 20 teeth per group in accordance with the guidance on µTBS testing of dental composite bonding [30].

Grouping of samples

Table 1: Materials used in the study

Eighty molar teeth were divided randomly into two main groups of 40 specimens each according to the types of restorative materials used in the study. Materials utilized in the study are illustrated in Table 1.

Group 1: restored by Admira Fusion resin composite and

Group 2: restored by Grandio resin composite

Each main group was further subdivided into two equal subgroups of 20 specimens each according to the application of hydrophobic resin coating (Heliobond).

Subgroup 1: Using Admira composite resin with its adhesive and then applying a hydrophobic resin coating (Heliobond).

Subgroup 2: Using Admira composite resin with its adhesive without the application of a hydrophobic resin coating (Heliobond).

Subgroup 3: Using Grandio Composite with its adhesive, then applying a hydrophobic resin coating (Heliobond).

Subgroup 4: Using Grandio Composite with its adhesive without the application of a hydrophobic resin coating (Heliobond).

Restorative procedures

Etching gel was applied to the exposed dentin surface with 37% phosphoric acid for 15 s and was subsequently rinsed away with air or water spray for 30 s, then gently air dried. A dual-cure universal adhesive in a single-dose delivery system (Futurabond DC, Voco, Germany) was chosen as the universal adhesive. The bonding agent was activated by pressing on the tab, forcing the liquids to combine within the package. The brush was used to perforate the foil and then mix the bonding agent. The bonding agent was then applied homogeneously to the exposed dentin surface and rubbed in for 20 s. The adhesive layer was gently dried with oil-free air for at least 5 s to remove any solvents, followed by light curing for 10s according to the manufacturer's instructions by a lightemitting diode curing unit (LED) with a light intensity of 1470 mW/cm² (3M Elipar Deep Cure-S LED Curing Light USA). Furthermore, for selected subgroups, application of a very thin layer of Heliobond with a microbrush on the exposed dentin surface was done. Apply an air blower to achieve an optimally thin layer, then lightcure for 10 s. Composite resin restorations were built up in two increments of 2 mm each. Each increment was light-cured for 40 s using a LED light-curing unit set at 1200 mW/cm² (Radiical, SDI Limited, Bays Water, Victoria, Australia) (Figure 2). The Tofflemire matrix (DDP, stainless steel, Pakistan) system was used to give the restoration its shape during resin composite packing. The specimens were sectioned longitudinally in mesio-distal and bucco-lingual directions across

Materials	Specification	Composition	Manufacturer	Batch number			
Admira Fusion	Nanohybrid ORMOCER** based resin	Matrix: Resin ORMOCER Filler: glass ceramics, Silicon oxide	Voco, Cuxhaven, German Service @voco.de	1934381			
Grandio	composite restorative material Nano hybrid Bis-GMA-Based resin composite	Nano filler, pigments. Filler: Inorganic filler content %: 84 (W/w) Resin matrix: based on dimeth-acrylates, contains Bis-GMA and TEGDMA *** Inorganic filler particles: Nano-sized silica)	Voco, Cuxhaven, German Service	1948567			
Futurabond DC	Dual-curing universal adhesive	filler content % (87% w/w-71.4vol) Organic acids, Bis-GMA, HEMA, BHT (butyle-hydroxy toluene; inhibitor), ethanol, fluorides, CQ, amine, catalysts	Voco, Cuxhaven, German Service	1924397			
Vococid etchant Heliobond	Etchant gel Hydrophobic resin coating	37% phosphoric acid. Silica. water bis-GMA, TEGDMA, initiators, stabilizers	Voco, Cuxhaven, German Service Ivoclar Vivadent, Schaan, Liechtenstein	1507285 U34134			
HEMA: 2-hydroxyethyl-methacrylate, BIS-GMA: Bisphenol-a-diglycidyl-ether-dimethacrylate, TEGDMA: Tri-ethylene-glycol-dimethacrylate, UDMA: Urethane dimethacrylate, BIS-EMA: Bisphenol A polyethylene glycol diether							

dimethacrylate

Open Access Maced J Med Sci. 2023 Aug 10; 11(D):115-123.



Figure 2: Light curing system

the bonded interface with a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) (Figure 3) to obtain resin-dentin beams (Figure 4) with a cross-sectional area of approximately 0.8 mm² measured with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan) (Figure 5)



Figure 3: Slow speed diamond saw

Testing procedures

The resin-dentin bonded beam was attached to the resin-dentin bonded beam holder with tetric-flow flowable composite (3M adhesive) (Figure 6) and tested under tension (Model 5565, Instron Co., Canton, MA, USA) (Figure 7) at 0.5 mm/min until failure. The μ TBS values were calculated by dividing the load at failure by the cross-sectional bonding area [31], [32]. The μ TBS values (MPa) of all beams from the same tooth were averaged for statistical purposes.

Statistical analysis

The data analysis was performed in several steps. Initially, descriptive statistics for each group resulted. A multi-factorial ANOVA was used to detect the effect of each variable (composite groups, adhesive



Figure 4: Resin-dentin beam

type, application mode, and resin coating). A one-way analysis of variance (ANOVA) was done between all subgroups. A student t-test was performed to detect significance between coat subgroups.



Figure 5: Digital caliper

Statistical analysis was performed using Assistant 7.6 statistics software for Windows (Campina Grande, Paraiba State, Brazil). $p \le 0.05$ is considered statistically significant in all tests.



Figure 6: Resin-dentin beam holder

Results

1. μ-TBS

The mean values and standard deviation of μ TBS measured in mega Pascal (MPa) for all composite groups as a function of adhesive type, application mode, and resin coating are summarized in Table 2 and graphically drawn in Figure 8.



Figure 7: Universal testing machine

Table 2: Comparison between total $\mu\text{-tensile}$ bond strength results (Mean ± SDs) as function of resin coat application

Variables	Mean ± SD	Tukey's rank	Statistics (p-value)				
Resin coating							
With C1	30.46 ± 6.7	A	0.0001*				
Without C2	23.95 ± 9.02	В					
Different letter in the same column indicating statistically significant difference ($n < 0.05$)							

2. Total effect of resin coating C (heliobond) on the composite resin used

Regardless of the different composite and adhesive types, there was a statistically significant difference (p < 0.05) among subgroups. It was found that subgroups with Resin coating, recorded a statistically significant (p < 0.05) higher μ TBS mean value (30.46 ± 6.7 MPa) than groups without Resin coating which recorded μ TBS mean value (23.95 ± 9.02 MPa), as indicated by a multi-factorial ANOVA followed by pairwise Tukey's post-hoc tests.



Figure 8: Column chart of total μ -tensile bond strength mean values as function of resin coat application

Interaction between different variables (Table 3 and Figure 9)

Filtek[™] Admira (A); it was found that the group with Resin Coating C was recorded statistically.

Table 3: Comparison of μ -tensile bond strength results (Mean ± SD) between all composite groups as function of adhesive type, resin coating with Etch and rinse application mode

Resin coating Heliobond (C)	With (C1)	Without (C2)	p-value			
Resin composite fillingA)						
Admira (A1)	$38.6^{A} \pm 4.7$	34.1 ^A ± 4.9	0.3919 ns			
Grandio (A2)	29.04 ^B ± 6.9	26.4 ^B ± 6.5	0.0918 ns			
ANOVA						
p-value	0.0002*	<0.0001*				
Different letters in the same column indicating statistically significant differences ($p < 0.05$). *Significant						

Different letters in the same column indicating statistically significant differences (p < 0.05). *Significant (p < 0.05) ns: Non-significant (p > 0.05).

non-significant (p > 0.05) higher μ TBS mean value than groups without Resin coating (C2), as indicated by the paired t-test in Figure 9.

Filtek[™] Grandio (B); it was found that the group with Resin Coating C was recorded statistically.

non-significant (p > 0.05) higher μ TBS mean value than groups without Resin coating (C2), as indicated by the paired t-test in Figure 9.



Figure 9: Column chart of μ TBS mean values for all composite groups as function of resin coating with Etch and rinse application mode

Discussion

Optimizing dentin bonding requires the development of adhesive solutions that boost microtensile strength. However, it is impossible to completely rule out the effects of marginal discoloration, recurrent caries, post-operative symptoms, and the durability of the resin composite restoration.

Admira Fusion (VOCO) primarily comprises ceramic polysiloxane, which shrinks less than other composite resins' organic dimethacrylate monomer matrix (1.25%). This kind of ormocer enhances appearance, biocompatibility, abrasion resistance, protection against caries, and lowers surface roughness and polymerization shrinkage stresses. Additionally, it does away with any worries about cytotoxicity linked to traditional monomers like BisGMA and TEGDMA. Especially when compared to composite resins based on methacrylates, this characteristic is a huge benefit [33].

Simplified universal adhesives that produce sticky contacts were used as permeable membranes [34]. Universal adhesives enable the transudation of dentinal fluid to the surface, where it collects as droplets once they have fully dried. But when a more hydrophobic solvent-free adhesive covering is utilized, degradations are less prevalent [35], [36]. It has been demonstrated that HC lengthens resin-dentin interfaces both in vitro and in vivo and decreases the likelihood of the bonds degrading hydrolytically [37], [38].

According to this research, the application of Heliobond may enhance the mean (TBS) for the adhesives when comparing hydrophobic resin coating versus noncoating. However, when Heliobond was skipped, the mean TBS for all groups may have decreased dramatically. This discovery emphasizes the potential protective function of a Heliobond at the adhesive contact during phosphoric acid dentin etching [39]. It is thought that the composition of the adhesives and the ensuing mechanical strength are better indicators of the strength of the initial connection than the adhesive's acidity. However, in the long run, water content at resin-dentin interfaces and the quantity of diffusion-induced water movement may be caused by the chemical makeup of universal adhesives [39].

The mean TBS increased as a result of the Heliobond application. A more densely packed hybrid layer with enhanced mechanical characteristics may have formed due to the possible increase in the adhesive layer's thickness [37]. Due to the increased hydrophobicity of the sticky layer, the HC also raised the mean TBS of SBU/self-etch mode (SE) and ABU/SE. The adhesive layer becomes less vulnerable to water deterioration and less permeable to water flow. By copolymerizing with the uncured adhesive surface, coating with a hydrophobic layer may link additional unsolved hydrophobic monomers to the adhesive interface, reducing the relative concentration of retained solvent and unreacted monomers and increasing the in-situ degree of conversion [37].

In spite of the circumstances for water storage, the monomer conversion process continues after the completion of the polymerization process because of the ongoing spread of free radicals [40]. The postpolymerization procedure may have been shielded by the hydrophobic layer. In another study, a 2-step mild selfetch adhesive (Opti Bond XTR, Kerr Co., Orange, CA, USA) produced mean TBS values that were higher after 6 months of water aging, in a similar range as the "golden standard" self-etch Clearfil SE Bond [41]. In addition to a putative chemical connection between a functional monomer molecule (GPDM, glycerol phosphate dimethacrylate) and calcium in hydroxyapatite, the existence of the hydrophobic resin step in the two-step self-etch adhesive may have contributed to this result.

The current study's findings agree with those of [2]. They examined the μ TBS of molars restored with contemporary restorative materials. They found that Admira filling material has the highest value when compared to the other restorative materials because it is based on ormocer technology, which should not be confused with glass ceramic fillers in conventional composites. In place of carbon, ormocers have a lengthy silicon "backbone" onto which side chains with carbon-carbon double bonds are grafted. A material of interest for use as a matrix for resin composites is ormocers because the larger size of the monomer molecule can minimize polymerization shrinkage and wear as well as reduce leaching of monomers.

Admira composite incorporates standard filler particles comprised of glass and ceramic. By replacing a significant portion of the organic resin matrix in traditional composites with these three-dimensional polymeric materials, polymerization shrinkage (1.25% by volume) is reduced. The findings of the present study concur with those of Gunwal *et al.* [42] They examined the fracture resistance, and mode of failure of premolars restored with nanohybrid composite, ormocer, and ceramic inlays. They found that ormocer had the best fracture resistance comparable to natural teeth. They suggested using ormocer as a filling material for posterior tooth restoration because it combines the surface characteristics of silicones, the toughness of organic polymers, and the strength of ceramic.

The outcome of the present investigation was also in agreement with those of Perdiou *et al.* [43], who investigated microtension stress resistance between nano-hybrid composite and ormocer restorations on posterior teeth. It was demonstrated that the ormocer is more resistant than the nanohybrid composite. It was claimed that the outcome of ormocer combined the toughness of organic polymers, the hardness, and the thermal stability of ceramics with the surface qualities of silicones to produce three-dimensionally cross-linked co-polymers with multi-polymerization and no remaining unreacted monomers.

The results of the current investigation were consistent with that of Margarit *et al.* [44], who found that teeth treated with ormocer had the highest μ TBS value, followed by those restored with nano-filled composite, and finally those restored with microhybrid resin composite. But in the study of Dina *et al.* [45], they compared the μ TBS of some MOD cavities in premolars that had been filled with various materials (ormocer, nano-filled, nanoceramic, and microhybrid composite), and they found that the teeth restored with nanoceramic composite had the best mean value of μ TBS. The varied resistance of the repaired teeth made of ormocer, nanofilled, nanoceramic, or microhybrid composite can be explained by the four materials' differing elastic moduli.

The findings of the current study were in contradcition with Klauer *et al.* [46], who examined the mechanical stability of bisphenol A-glycidyl methacrylate (Bis-GMA) and Ormocer-based resin composites. They found that Admira Fusion is a promising Bis-GMA-free and Ormocer-based material, but it does not exhibit comparable mechanical performance to traditional Bis-GMA-containing resin composites. They utilized the teeth that have undergone endodontic treatment, which may account for the variation in their outcomes.

Conclusion

Within the limitations of this study, it is concluded that an extra hydrophobic layer coating improved the performance of the μ TBS of the adhesive systems utilized with the new BIS-GMA-free versus BIS-GMA-containing composite resin.

Recommendations

In vitro studies should be followed by in vivo studies for the application of this coating in different dental applications.

Declarations

Ethics approval and consent to participate

Approval from the local ethics committee (#06\11\22 NUB-MREC).

Availability of data and materials

The authors announce that the data supporting the results of this study are existing within the article.

Authors' Contributions

LMS performed the study design. LMS did μ TBS test. LMS prepared the manuscript. LMS read and approved the final manuscript.

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