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Article

Bond Strength of Low-Viscosity Bulk-Fill Composite and Adhesives Using 10-Methacryloyloxydecyl Dihydrogen Phosphate (10-MDP)

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Abstract: Purpose: to evaluate the bond strength of low-viscosity bulk-fill composite and adhesives using 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). Materials and Methods: iBOND by Kulzer, Prime&Bond elect by DENTSPLY Caulk, TOKUYAMA UNIVERSAL BOND II, Tokuyama Dental Corporation, and Adper Easy Bond Self-Etch Adhesive, as well as one 10-MDP-free adhesive (Xeno IV DC, Dentsply Sirona) were applied to polished, air-abraded surfaces of randomly allocated Filtek™ Bulk Fill Flowable Restorative blocks. 3M™ Filtek™ Universal Restorative was then applied in layers after the adhesives. Using a hard-tissue microtome, each multilayer composite block was cut into stick specimens. Microtensile bond strength was measured on half of the groups (immediate group), while the remaining groups were matured in a thermocyling machine for 5000 cycles before having their microtensile bond strength tested (aged group). Scanning electron microscopy was used to assess the adhesive contact (SEM). Light microscopy was used to observe failure modes. Levene's test, ANOVA, Welch's ANOVA, Tukey's test, and the Z-test were used to analyze the results as necessary (significance: p 0.05). Results: The binding strength between the 10-MDP-containing adhesives and the 10-MDP-free glue varied significantly across all groups. In all glue groups, aging considerably reduced the binding strength. The binding strength and endurance of the 10-MDP-containing adhesives did not differ significantly from one another. Conclusion: When applied to the air-abraded Filtek™ Bulk Fill Flowable Restorative composite surface, 10-MDP-containing adhesives perform better than 10-MDP-free adhesives. The binding strength was unaffected by the 10-MDP-containing adhesives' chemical makeup. The bond strength endurance of adhesives containing 10-MDP decreases with age.

Keywords: keyword 1; keyword 2; keywords: low-viscosity bulk fill composite; universal adhesives; self-etch adhesive; 10-MDP; aging; bond strength

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1. Introduction

In everyday clinical practice, light-cured resin composites (RBCs) are the first option of restorative materials since minimally invasive and cosmetic treatments are preferred in dentistry. 14 During the restorative method, conventional composites should be stacked incrementally, and the oxygen-inhibiting layer (OIL) on the uppermost composite surface is often enhanced the copolymerization of successive composite layers. Bulk-fill resin composite (BFRC), which permits an increase thickness of 4-5 mm, was created as a way to simplify the time-consuming and technically delicate application process. An alternate photoinitiator method and freshly synthesized monomers linked to stress-reduction technology can be found in BFRCs.[1,2]

There are two types of BFRCs: flowable (low viscosity) and full-body (high viscosity), each having a unique therapeutic application process. Yet, since universal resin composites must be employed as a cap on top of the restoration because low-viscosity BFRCs are mostly used as dentin-replacement materials [1,3]

Some clinical circumstances lead to the loss or contamination of the oil and it may affect how a fresh composite layer is applied. To promote adhesion between both the composite layers in these specific situations, the damaged composite surface must be reactivated by roughening and/or wetting the surface. As a quick fix, this technique can be applied. [3,4]

The strength and longevity of adhesion are crucial for achieving interface stability. Programmes emphasize the value of physical surface treatments and support the effectiveness of chemical conditioning techniques [3.4], but they come to different findings on the best regimen. Although a fresh composite surface is much more activation-friendly than an old, crumbling one, there is little information on how to activate a flowable bulkfill composite surface using adhesives that contain 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), and the longevity of the interface is also in doubt. Due of their versatility in use and multimodality, universal adhesives are well-liked. Moreover, since they're self-etch adhesives, the application method is simpler. These adhesives' cutting-edge technology enables one-step bonding, priming, and etching with minimal technical sensitivity [2]. The precise composition and complexity of universal and self-etch adhesives are designed to create a stable and adequate bond strength, but their water sorption and product-dependent effectiveness are causes for worry. Several or more acidic functional molecules are present in universal and self-etch adhesives, which improve conditioning and chemical interaction. One of the most adaptable functional monomers is 10-MDP; it has a very high potential for adhesion to a wide range of substrates, including metals, lithium disilicate, zirconia ceramics, and dental hard tissues, and it appears to be essential for self-etch adhesives to reach a stable bond strength [3,5].

It has been studied how well universal adhesives perform under various protocols. 16 In order to examine the performance of four 10-MDP-containing adhesives on the low-viscosity bulk-fill composite surface and the dependability of the technique after aging, the microtensile bond strength (TBS) was measured [5,6].

We applied adhesives containing 10-MDP, aged the specimens using thermocycling, and employed air abrasion as the industry-standard mechanical surface treatment [4] (in accordance with ISO/TS 11405:2015). Three theories were investigated: The effectiveness of 10-MDP-containing adhesives is unaffected by aging, and there is no discernible difference in the bond strength durability when contrasting 10-MDP-containing adhesives with 10-MDP-free adhesives. [1] There is no appreciable difference between the bond strengths of 10-MDP-containing adhesives with variable composition. [2].

2. Materials and Methods

Study Materials

Five different adhesives - Xeno IV DC (XEN, Dentsply Sirona), iBOND by Kulzer, Prime&Bond elect (DENTSPLY Caulk), TOKUYAMA UNIVERSAL BOND II, Tokuyama Dental Corporation and Adper Easy Bond Self-Etch Adhesive (3M ESPE) - were applied on the \surface of Filtek™ Bulk Fill Flowable Restorative composite (Dentsply \Sirona; Konstanz, Germany) as the substrate. 3M™ Filtek™ Universal Restorative composite was used to finish the layering process. Table 1 contains information about the materials' description, composition, and producers. Except for HB, which functioned as a hydrophobic adhesive control and was devoid of solvent and acidic monomers, all adhesives contained 10-MDP or its derivatives.

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Composition and manufacturers of resin composite and adhesive materials

	Material	Code	Manufacturer	Components
1	3M™ Filtek™ Universal Restorative		3M™ ESPE	AUDMA, AFM, diurethane-DMA, and 1,12-dodecane-DMA. non-agglomerated/non-aggregated silica filler 20nm, a non-agglomerated/non-aggregated 4 to 11nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20nm silica and 4 to 11nm zirconia particles), and a ytterbium trifluoride filler consisting of agglomerated 100nm particles.
2	Filtek™ Bulk Fill Flowable Restorative		3M™ ESPE	bisGMA, UDMA, bisEMA and Procrylat resins, zirconia/silica 0.01 to 3.5µ and ytterbium trifluoride filler with a a with a particle size range of particle sizes from 0.1 to 5.0µ
3	Xeno IV DC			Xeno® IV Adhesive: Mono-, Di- and Trimethacrylate resins; PENTA (dipentaerythritol penta acrylate monophosphate); Photoinitiators; Stabilizers; Cetylamine hydrofluoride; Acetone; Water Self Cure Activator: Mono- and Di-methacrylate Resins; Catalyst; Photoinitiators; Stabilizers; Acetone; Water
4	iBOND Universal		Kulzer	4-META, MDP, Methacrylates, Acetone, Water
5	Prime&Bond elect		Dentsply Sirona	Prime&Bond elect Adhesive: Mono-, di- and trimethacrylate resins; PENTA (dipentaerythritol penta acrylate monophosphate); Diketone; Organic phosphine oxide; Stabilizers; Cetylamine hydrofluoride; Acetone; Water Self Cure Activator: Mono- and Di-methacrylate Resins; Catalyst; Photoinitiators; Stabilizers; Acetone; Water
6	TOKUYAMA UNIVERSAL BOND II		Tokuyama Dental Corporation	Phosphoric acid monomer, Bisphenol A di(2-hydroxy propoxy) dimethacrylate (Bis-GMA), Triethylene glycol dimethacrylate (TEGDMA), 2-Hydroxyethyl methacrylate (HEMA), MTU-6 (thiouracil monomer), Silane coupling agent, Peroxide, Borate catalyst, Acetone, Ethanol and Purified water.
7	Adper Easy Bond Self-Etch Adhesive		3M™ ESPE™	2-hydroxyethyl methacryate (HEMA), Bis-GMA, Methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate Methacrylate, functionalized Polyalkenoic acid (Vitrebond™ Copolymer), Finely dispersed bonded silica filler with 7 nm primary particle size, Ethano,l Water, Initiators based on camphorquinone Stabilizers

Specimen Preparation for µTBS Measurements

A specially constructed Teflon mold measuring 10 mm by 10 mm by 7 mm was used to create SDR blocks. The bulk-fill technique was used to apply layers that were four millimeters thick (Figure 1). In a Scheu LC-6 light oven (Iserlohn, Germany) fitted with various light tubes (three UVA, three blue light, with maxima of 370 nm and 450 nm, respectively), each increment was polymerized for 180 s.

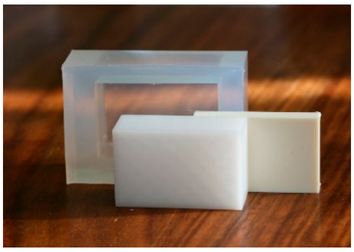

Figure 1. Custom made Teflon mold.

Surface treatment of Filtek™ Bulk Fill Flowable Restorative blocks

Using 400, 800, and 1200 grit silicon-carbide abrasive papers and water cooling, the adhesive surface of the Filtek™ Bulk Fill Flowable Restorative blocks was polished using a polishing machine (Struers LaboPol35; Rdovre, Denmark) at 300 rpm for 30 seconds. After polishing, the blocks underwent a 10-minute ultrasonic cleaning to remove any remaining abrasive materials. An intraoral sandblaster (Bio Art, Dentmark, Dental Equipment) was used to sandblast 50-m Al2O3 (BDSI, Dental Equipment & Consumables) onto the polished Filtek™ Bulk Fill Flowable Restorative blocks for 10 seconds at a distance of 10 mm under 2.5 bar of pressure. This was followed by 90 seconds of washing and 90 seconds of drying with an air-water syringe. Before being adhesively attached to TEC, the cured and polished blocks were allowed to dry out at room temperature for 24 hours.

Application of adhesives (Figure 1)

A thin layer of each adhesive was placed, in accordance with the manufacturer's instructions, to a randomly selected sandblasted SDR surface after 24 hours. Table 2 provides a summary of adhesive application methods. With the use of an oil-free airwater syringe, the adhesives were dried. With a dental light-curing device (Elipar™ DeepCure LED Curing Light) set to a high-mode curing program (1470 mW/cm2), all adhesives were light-cured.

Application mode of adhesives

Xeno IV DC	iBOND Universal	Prime&Bond elect	TOKUYAMA UNIVERSAL BOND II	Adper Easy Bond Self-Etch Adhesive
Duration of application (s)	20 seconds	Condition enamel for at least 15 seconds and dentin for 15 seconds or less.	25-seconds	20 seconds
Motion	The liquid needs to be agitated in the cavity to make sure that consumed monomers are removed and fresh monomer come in contact with the tooth surface	Thoroughly wet all the tooth surfaces. Agitate the applied adhesive for 20 seconds. Re-wetting of the microbrush may be required in order to coat the preparation for the full 20 seconds.	A and B into the same dimple of disposable mixing well and mix. Apply the mixed bond.	Apply using a rubbing motion
Drying time	Because of the needed water activation of the acidic groups of self-etch adhesives, these products contain, in addition to alcohol or ethanol, plenty of water that must be removed from the adhesive layer by sufficient air-drying prior to polymerisation.	Rinse conditioned areas thoroughly for at least 15 seconds. Remove rinsing water completely by blowing gently with an air syringe or by blot drying with a cotton pellet.	no need to wait	5 seconds
Polymerization time	Short curing time of only 10 seconds.	Cure for 10 seconds using a curing light, spectral output containing 470nm, minimum light output at least 550mW/cm2.	no need to light cure	10 seconds

Application of universal composite

The Filtek™ Bulk Fill Flowable Restorative blocks were reinserted into the Teflon mold after adhesives had been used, and 3M™ Filtek™ Universal Restorative composite repair was made in accordance with the manufacturer's specifications. Each layer of the 3M™ Filtek™ Universal Restorative composite was polymerized for three minutes in a Scheu LC-6 light oven after being applied in 2-mm increments. Following 24 hours, the restored block was cut in two with a hard-tissue microtome Bluedent India) equipped with a diamond saw while being cooled by water. Stick-shaped specimens measuring 1 x 1 x 15 mm were the result. 90 non-trimmed sticks from each group were

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separated into two groups by drawing 30 at random. The first was subjected to group TBS measures, while the second was aged.

Aging of the interface

The thermocycling machine (Scalibra Calibration Lab., Skjetten, Norway) was used to age the second group of slices (1 mm x 1 mm x 15 mm) for 4000 cycles at 5-55oC with a 30-s dwell period. The sticks' TBS was determined after age. Figure 2 displays the experimental groups according to the adhesives used and the aging procedure.

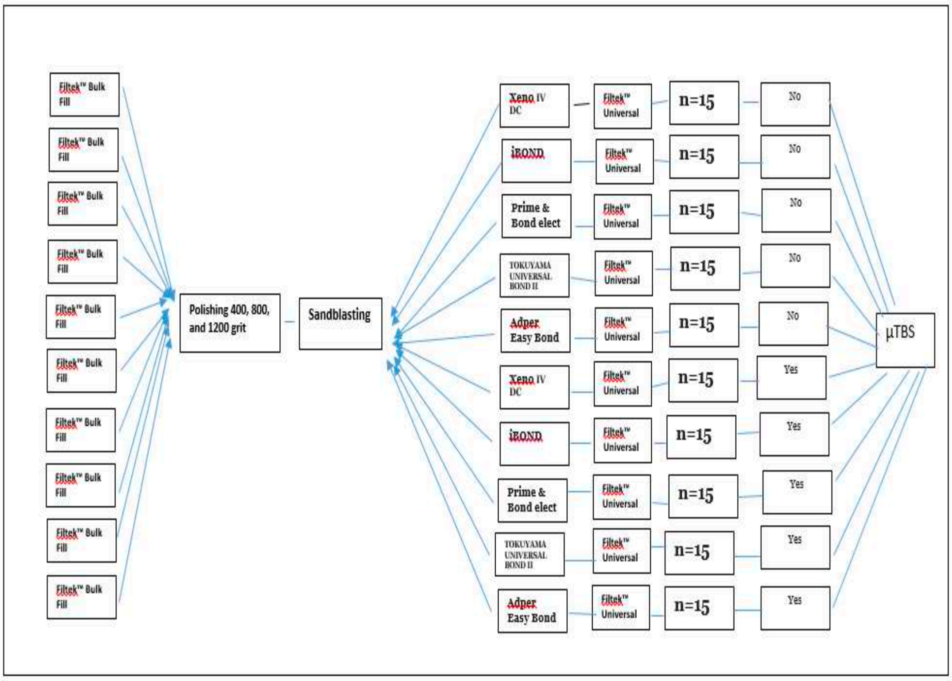

Fig 2: flowchart test groups filtek™ bulk fill flowable restorative, 3m™ filtek™ universal restorative , XENO IV DC, Ibond universal, Prime&Bond elect, Tokuyama Universal Bond II, Adper Easy Bond Self-Etch Adhesive

µTBS Measurements

A digital calliper was used to measure the width and thickness of each sample at three distinct locations. The average width and thickness were determined using these measurements.

The aged and unaged sticks were fastened to a metallic cuvette with an active grip notch. The cuvette was put inside a mechanical analyzer with a 2-kN load cell (Instron 5566; Norwood, MA, USA). A 1 mm/min crosshead speed was chosen. By dividing the measured load (N) by the cross-sectional area, the TBS was computed (mm2).

Detection of the Failure Mode

To identify the type of failure, all fragmented surfaces were examined with a stereo lightmicroscope (Leica 7.5 Mz, Microsystems).

Ltd. Business Unit SM, Heerbrugg, Switzerland) at a 45X magnification. The failures were separated into two categories: cohesive failures that happened within the Bulk Fill Flowable Restorative or Universal Restorative composite and adhesive failures that occurred at the contact between the Bulk Fill Flowable Restorative and Universal Restorative composite.

Scanning Electron Microscopy (SEM)

A dual-beam focused ion-beam Scios 2 scanning electron microscope (JSM-5910LV, Tokyo, Japon) was used to examine the surface morphology of the samples under low accelerating voltage (2 keV). The structure and morphology of insulating samples (such as biological samples, aerogels, polymers, etc.) without a gold layer coating was studied using low energy and a short working distance (2 mm). Secondary electrons, backscattered electrons, or a combination of both types of signals can be distinguished from and collected by a specialized detector, referred to as an in-lens detection system. Discrepancies and unevenness are not concealed by the gold sputter-coating-free approach, which is an advantage.

Statistical Analysis

Using Levene's test, the homogeneity of variability was examined. The means of the groups were compared using a one-way ANOVA for data with uniform variance. Welch's ANOVA was used to compare the means of the groups for data with homogeneous variance. For pairwise comparisons, we next applied the proper post-hoc test, such as the Tukey's honestly significant masked (HSD) test or the Tamhane test. To identify adhesive or cohesiveness percentages that were distinct from 50%, binomial testing were used. In order to contrast the rates of adhesive cracks between A two-sample Z-test for proportions was used for immediate and aged cases. All tests were performed using IBM's SPSS Statistics 27 software, with the exception of the two-sample Z-test of proportions were computed in R. 20.

RESULTS

μTBS Results

In Figure 3, the TBS data are displayed. The tested treatments had a mean TBS that ranged from 36.4 MPa to 46.6 MPa. In all groups, there was a statistically significant difference in TBS between the 10-MDP-containing and 10-MDP-free adhesives (p 0.05). In all adhesive groups, aging significantly decreased TBS (p 0.05). The aged groups of 10-MDP-containing adhesives had significantly higher variations in TBS (p 0.05), which were related to wider ranges and lower minima (Figure 3). TBS did not significantly differ between the old and immediate groups for the adhesives containing 10-MDP (p 0.05).

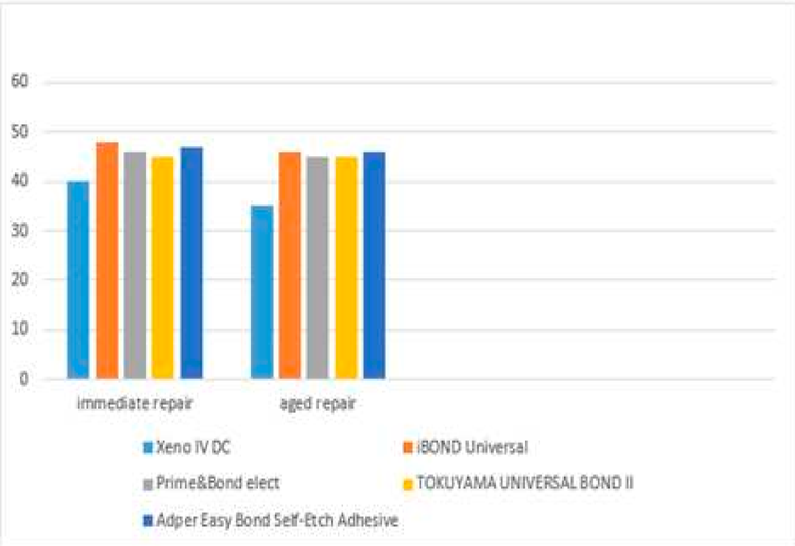

Fig 3: Microtensile bond strength results according to the tested adhesives and aging protocol (box plots are shown.

Failure Mode Analysis

Results for failure modes are shown in Figures 4 and 5. The immediate groups with adhesive containing 10-MDP experienced a much greater rate of adhesive failure: 98% for Adper Easy Bond Self-Etch Adhesive, 92.1% for Tokuyama Universal Bond II, 87.3% for PBE, and 64.9% for iBOND. Nonetheless, cohesive failure (56.8%) was the most prevalent failure category for Xeno IV DC. The percentages of cohesive failure were often much higher in the older groups: 84.6% for the Adper Easy Bond, 81% for the PBE, 82% for the TBF II, and 72.9% for the HB. In contrast, the elderly TUB group (81%) was mostly affected by adhesive failures.

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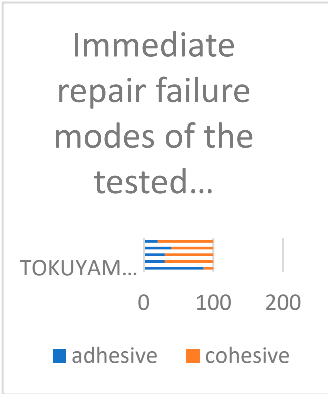

Figure 4. Immediate repair failure modes.

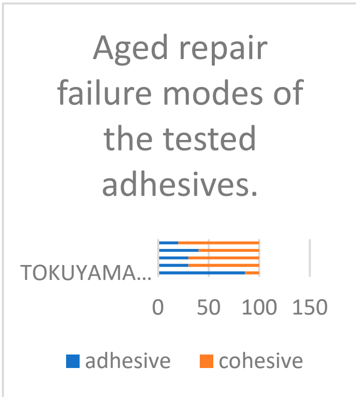

Figure 5. Aged repair failure modes of the tested adhesives of the tested adhesives.

SEM

Depending on the applied adhesives and aging technique, Figure 6 displays the SEM results. Each image has a left and right half, with the left half displaying SDR and the right half TEC. In contrast to the M™ Filtek™ Universal Restorative, which has a smoother surface, the scanning electron micrographs of the Filtek™ Bulk Fill Flowable Restorative composite indicate big filler particles to have an uneven size distribution. Both the fresh and aged samples have clearly defined interfaces.

DISCUSSION

In this study, we assessed the TBS of four 10-MDP-containing adhesives to a low-viscosity bulk-fill resin composite and looked at the bond strength both before and after a thermocycling regimen Ibond, Prime&Bond elect [PBE], TOKUYAMA UNIVERSAL BOND II (TUB), and Adper Easy Bond (AEB). In order to address the shortcomings of multistep etch-and-rinse adhesives used during final restorations and to provide chemical adherence in certain clinical circumstances, self-etch and universal adhesives were developed[7,8]. The chemical processes significantly alter the chemical makeup of self-etch adhesives and greatly enhance adhesion quality. Consequently, interactions between the various adhesive components, application procedures, and substrate surface quality affect the clinical performance and effectiveness of self-etch adhesives [9–11].

Hydrophilic and hydrophobic molecules are combined in simplified adhesives, but the purity and concentration of the monomers varies depending on the product, which significantly impacts the binding strength and longevity [8].

Although the long-term efficacy of universal adhesives on dentin and enamel has previously been studied [9.2, 13] there is a dearth of information about the bond strength of adhesives containing 10-MDP on flowable bulk-fill composite surfaces. The 10-MDP-containing universal and self-etch adhesives examined in this study may adhere to a wide range of substrates, including silica, metal oxides, zirconia, and resin monomers. 35 The linking molecules may provide an effective protective zone against biodegradation at the adhesive contact in the form of a stable nanolayered structure [14,15].

Since HB is a bonding agent with no acidic functional monomer, we utilized it as a control. Adhesives using acidic functional monomers include organic solvents (such as alcohol or acetone) that lessen the mixture's viscosity and aid the monomers in penetrating surface imperfections. Due to the solvent and the monomers' excellent miscibility, solvent can be retained in the interfacial layer after the adhesive layer has dried. The solvent that is still present may have an impact on how well SDR and TEC adhere.

With the solvent-free HB glue, this phenomena is not seen. Based on their molecular mobility, the faults of the sandblasted surface of the composite are filled with a slightly viscous monomer mixture.

The substrate for the tested adhesives was Filtek™ Bulk Fill Flowable Restorative composite. Filtek™ Bulk Fill Flowable Restorative has a high degree of conversion and had a modest filler load with particles made of barium, aluminum, and silica that were different sizes (0.01 to 3.5μ) [16.17]. These big particles may be useful to resin bonding agents as a retentive region. 3 Chemical and mechanical components both have an impact on adhesion at the composite-composite interface [18,19]. Thus, the composite resin surfaces were ground with silicon carbide disks (up to 1200 grit), followed by air abrasion with 50-m Al2O3 particles, before adhesive was applied. This approach is applicable to clinical scenarios in which an immediate correction is necessary owing to failure after completing an RBC restoration. A brand-new composite surface is an idealized surface devoid of hydrolysis or degradation traces. Unreacted monomers give the intermediate agent the C=C needed to generate C-C covalent bonds. Furthermore, functional monomers join with the fillers to raise the cohesive strength of the composite substrate by strengthening the bonds between them [3,18].

The 10-MDP-containing adhesives under investigation here have TBS that are consistent with those provided by Ahmed et al., Yilmaz et al., [19] and Sismanoglu et al. [20].

These researchers discovered strong adhesion to the flowable bulk-fill composite, and this is in line with our SEM findings. Also, in the immediate groups, the TBS of all tested 10-MDP-containing adhesives was much higher than with the control adhesive, which is similar with the findings of an earlier investigation. 10 Similar to the conclusions of Isolan et al.[21] and Suarez et al.6, although in contrast to results from a previous investigation, the composition of the 10-MDP-containing adhesives varied in this study but did not produce significantly different TBS [22]. We therefore agreed with our initial theory.

The relatively thin Adper Easy Bond adhesive layer seen in scanning electron micrographs is consistent with the application of Adper Easy Bond, which required a brief burst of maximum air pressure. The application protocol's air-thinning step may have an impact on the bond layer thickness, but the filler's presence doesn't seem to have much of an impact. Before to applying adhesive, silanization has been recommended as a separate priming process to enhance wetting and Bonding.

According to a research hypothesis, silane inclusion in adhesives enhances wetting and sticking ability [23], much like a separate silanization phase [10] The process may be made simpler by include silane in the adhesive agent, but other factors, like as the bonding agent's composition and pH, may also have an impact on how well it affects TBS [24]. Adhesives with silane (PBE and TUB) or without silane (iBOND and AEB) demonstrated comparable TBS in the young and old groups regardless of the silane level. These results concur with those of Moritake et al., [15] Suzuki et al., [25], and Ouchi et al. [17] The stability of silane may be harmed by the acidic pH of PBE and TUB, leading to a changed chemical formula with a decreased priming capability.

2-hydroxyethyl methacrylate (HEMA) may function in part as a solvent to prevent phase separation and it may enhance surface wetting when used as an ingredient of dental adhesives. The formation of the 10-MDP interfacial nanolayer and high water uptake [26] have both been linked to it, as well as an inhibitory effect on polymerization [27]. Only one of the adhesives we tested, GP-

Premio Bond, is HEMA free, however it did not have a considerably higher TBS than the other adhesives that contained 10-MDP. This discovery differs from those made by Hardan et al. [1]

PBE comprises Vitrebond copolymer (VCP), a self-adhesive glass-ionomer-based polyalkenoic acid copolymer that has demonstrated outstanding bonding performance. 23 In line with a prior study, SU did not improve the repair bond strength when compared to the other universal or self-etch adhesives [28]. The interactions between SU components, such as the high-molecular-weight polyalkenoic copolymer, may make it difficult for 10-MDP to adhere to the same substrate, which is one argument that might be put up [29]. The polyalkenoate reaction may also be hampered by the components of the resin [25]. Thermocycling is an effective technique for mimicking the effects of hydrolysis, water sorption, and heat stress; as a result, it is excellent for evaluating how long a bonded interface will last. The cross-linked matrix deterioration, monomer leaching, hydrolysis of the resin polymer and resin-filler interface, microcrack development, and deterioration of the bonded resin interface weaken the repair bond. [3,25,30] According to Moritake et al. [15], Altinci et al. [31], and Zhang et al. [32], as well as Altinci et al. [31] and other studies, the bond strength was considerably weaker in the elderly than in the proximate groups in our study. We therefore disproved our second hypothesis. The limited hydrolytic stability of self-etch adhesives is consistent with this result. HEMA, silane, or hydrophilic substances with hydroxyl or phosphate groups may hasten the degradation of the bonded interface [33]. Notwithstanding this fact, the adhesive groups comprising 10-MDP had a much greater TBS than those made with HB. While it has been proposed that the hydrophobic resin layer act as a protective layer to lessen the hydrophilic degradation of universal adhesives [34], the reduction in TBS of HB was also notable. The TBS reduction for TBF II was 9%, SU was 9%, TUB was 10%, GP was 8%, and HB was 13%. Independent of composition or application method on the bulk-fill resin composite surface, these alterations show a comparable deterioration trend in all adhesive groups [35].

With the exception of Xeno IV DC, there was a bigger proportion of adhesive fractures in the proximate groups, demonstrating the similarity of the 10-MDP-containing adhesives. With the exception of TUB, Altinci et al 3 and Moritake et al [15] both reported that the cohesive fracture kind was the primary kind found after age. The existence of the hydrophilic amide methacrylate component may be the cause of this variation. Although scanning electron micrographs showed gap-free, well-integrated, tight interfaces in all groups, the hydrolytic degradation and softening of the resin matrix, as well as the loosening of the filler particles in parallel with interface disintegration, may be the cause of the majority of cohesive fractures [36,37].

Our use of TBS to assess binding strength is in line with earlier research [38,39]. Nonetheless, there are well-known drawbacks of in-vitro research. Therefore, additional research should be done to assess the impact of prolonged aging or the durability of the binding strength of multiple-layered adhesives.

CONCLUSIONS

The TBBS to a low-viscosity bulk-fill resin composite is unaffected by the makeup of adhesives containing 10-MDP. The bond strength of 10-MDP-containing and 10-MDP-free adhesives decreases with aging. In the Filtek™ Bulk Fill Flowable Restorative - Universal Restorative interface, 10-MDP-containing adhesives appear to be more efficient and long-lasting than non-solvated, 10-MDP-free adhesives.

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References

1. Effect of Different Application Modalities on the Bonding Performance of Adhesive Systems to Dentin: A Systematic Review and Meta-Analysis. Hardan L, Bourgi R, Cuevas-Suárez CE, Devoto W, Zarow M, Monteiro P, Jakubowicz N, Zoghbi AE, Skaba D, Mancino D, Kharouf N, Haïkel Y, Lukomska-Szymanska M. Cells. 2023 Jan 3;12(1):190.
2. Do Universal Adhesives Benefit from an Extra Bonding Layer? Ahmed MH, De Munck J, Van Landuyt K, Peumans M, Yoshihara K, Van Meerbeek B.J Adhes Dent. 2019;21(2):117-132.
3. Long-Term In Vitro Adhesive Properties of Two Universal Adhesives to Dentin. Hurtado A, Fuentes V, Cura M, Tamayo A, Ceballos L. Materials (Basel). 2023 Apr 28;16(9):3458.
4. Effect of Different Application Modalities on the Bonding Performance of Adhesive Systems to Dentin: A Systematic Review and Meta-Analysis. Hardan L, Bourgi R, Cuevas-Suárez CE, Devoto W, Zarow M, Monteiro P, Jakubowicz N, Zoghbi AE, Skaba D, Mancino D, Kharouf N, Haïkel Y, Lukomska-Szymanska M. Cells. 2023 Jan 3;12(1):190.
5. Bond Strength of Universal Adhesives to Dentin: A Systematic Review and Meta-Analysis. Hardan L, Bourgi R, Kharouf N, Mancino D, Zarow M, Jakubowicz N, Haikel Y, Cuevas-Suárez CE. Polymers (Basel). 2021 Mar 7;13(5):814.
6. Is the presence of 10-MDP associated to higher bonding performance for self-etching adhesive systems? A meta-analysis of in vitro studies. Fehrenbach J, Isolan CP, Münchow EA. Dent Mater. 2021 Oct;37(10):1463-1485.
7. Bonding Performance of Universal Adhesives: An Updated Systematic Review and Meta-Analysis. Cuevas-Suárez CE, da Rosa WLO, Lund RG, da Silva AF, Piva E. J Adhes Dent. 2019;21(1):7-26.
8. Which self-etch acidic composition may result in higher dental bonds at the long-term? A network meta-analysis review of in vitro studies. Fehrenbach J, Lacerda-Santos R, Machado LS, Miotti LL, de Carvalho FG, Münchow EA. J Dent. 2022 Nov;126:104283.
9. Adhesive interfacial characteristics and the related bonding performance of four self-etching adhesives with different functional monomers applied to dentin. Wang R, Shi Y, Li T, Pan Y, Cui Y, Xia W. J Dent. 2017 Jul;62:72-80.
10. Effect of the demineralisation efficacy of MDP utilized on the bonding performance of MDP-based all-in-one adhesives. Fujita Nakajima K, Nikaido T, Francis Burrow M, Iwasaki T, Tanimoto Y, Hirayama S, Nishiyama N. J Dent. 2018 Oct;77:59-65.
11. Development of MDP-based one-step self-etch adhesive--effect of additional 4-META on bonding performance. Iwai H, Fujita K, Iwai H, Ikemi T, Goto H, Aida M, Nishiyama N. Dent Mater J. 2013;32(1):1-9.
12. Which self-etch acidic composition may result in higher dental bonds at the long-term? A network meta-analysis review of in vitro studies. Fehrenbach J, Lacerda-Santos R, Machado LS, Miotti LL, de Carvalho FG, Münchow EA.J Dent. 2022 Nov;126:104283.
13. Are universal adhesives in etch-and-rinse mode better than old 2-step etch-and-rinse adhesives? One-year evaluation of bonding properties to dentin. Wendlinger M, Pomacóndor-Hernández C, Pintado-Palomino K, Cochinski GD, Loguercio AD. J Dent. 2023 May;132:104481.
14. Influence of a hydrophobic resin coating on the bonding efficacy of three universal adhesives. Muñoz MA, Sezinando A, Luque-Martinez I, Szesz AL, Reis A, Loguercio AD, Bombarda NH, Perdigão J. J Dent. 2014 May;42(5):595-602.
15. Effect of active application adhesives.Moritake N, Takamizawa T, Ishii R, Tsujimoto A, Barkmeier W, Latta M, Miyazaki M. Oper Dent 2019;44:188–199.
16. Bond Durability of Two-Step HEMA-Free Universal Adhesive. Tsujimoto A, Fischer NG, Barkmeier WW, Latta MA. J Funct Biomater. 2022 Aug 29;13(3):134.
17. Comparison of enamel bond fatigue durability of universal adhesives and two-step self-etch adhesives in self-etch mode. Tsujimoto A, Barkmeier WW, Hosoya Y, Nojiri K, Nagura Y, Takamizawa T, Latta MA, Miyazaki M. Am J Dent. 2017 Oct;30(5):279-284.
18. Comparison between universal adhesives and two-step self-etch adhesives in terms of dentin bond fatigue durability in self-etch mode. Tsujimoto A, Barkmeier WW, Takamizawa T, Watanabe H, Johnson WW, Latta MA, Miyazaki M. Eur J Oral Sci. 2017 Jun;125(3):215-222.
19. Effects of different universal adhesives and surface treatments on repair bond strength between resin composites. Yilmaz F, Yazkan B, Herguner Siso S. J Esthet Restor Dent 2022;34:1068-1076 .
20. Efficacy of different surface treatments and universal adhesives on the microtensile bond strength of bulk-fill composite repair. Şişmanoğlu S, Gürcan AT, Yıldırım-Bilmez Z, Gümüştaş B. J Adhes Sci Technol 2020;34:1115–1127
21. Effect of different adhesive protocols on bond strength between composite resinsfor indirect use and repair materials. solan CP, Moreira AG, Ribeiro JS, Nakanishi L, Cuevas-Suárez CE. J Adhes Sci Technol 2020;34:67–75.

22. Effect of double-layer application on bond quality of adhesive systems. Fujiwara S, Takamizawa T, Barkmeier WW, Tsujimoto A, Imai A, Watanabe H, Erickson RL, Latta MA, Nakatsuka T, Miyazaki M. J Mech Behav Biomed Mater. 2018 Jan;77:501-509.
23. Comparison of enamel bond fatigue durability between universal adhesives and two-step self-etch adhesives: Effect of phosphoric acid pre-etching. Suda S, Tsujimoto A, Barkmeier WW, Nojiri K, Nagura Y, Takamizawa T, Latta MA, Miyazaki M. Dent Mater J. 2018 Mar 30;37(2):244-255.
24. Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching. Lührs AK, Guhr S, Schilke R, Borchers L, Geurtsen W, Günay H. Oper Dent. 2008 Mar-Apr;33(2):155-62.
25. Bond durability of universal adhesives to intact enamel surface in different etching modes. Suzuki M, Takamizawa T, Hirokane E, Ishii R, Tsujimoto A, Barkmeier WW, Latta MA, Miyazaki M. Eur J Oral Sci 2021;129:12768
26. Comparison of bond strength of universal adhesives using different etching modes: A systematic review and meta-analysis. Chen H, Feng S, Jin Y, Hou Y, Zhu S. Dent Mater J. 2022 Feb 1;41(1):1-10.
27. Microtensile bond strength to dentin and enamel of self-etch vs. etch-and-rinse modes of universal adhesives. Cruz J, Sousa B, Coito C, Lopes M, Vargas M, Cavalheiro A. Am J Dent. 2019 Aug;32(4):174-182.
28. Dentin Permeability and Nanoleakage of Universal Adhesives in Etch-and-rinse vs Self-etch Modes. Cruz J, Silva A, Eira R, Sousa B, Lopes M, Cavalheiro A. Oper Dent. 2021 May 1;46(3):293-305.
29. How "Universal" is Adhesion? Shear Bond Strength of Multi-mode Adhesives to Enamel and Dentin. Jacker-Guhr S, Sander J, Luehrs AK. J Adhes Dent. 2019;21(1):87-95.
30. Effects of the Contents of Various Solvents in One-step Self-etch Adhesives on Shear Bond Strengths to Enamel and Dentin. Ageel FA, Alqahtani MQ. J Contemp Dent Pract. 2019 Nov 1;20(11):1260-1268.
31. Altinci P, Mutluay M, Tezvergil-Mutluay A. Repair bond strength of nanohybrid composite resins with a universal adhesive. Acta Biomater Odontol Scand 2018;4:10–19
32. Defying ageing: An expectation for dentine bonding with universal adhesives? Zhang Z yi, Tian F cong, Niu L na, Ochala K, Chen C, Fu B ping, Wang X yan, Pashley DH, Tay FR. J Dent 2016;45:43–52.
33. The effect of 6-month water storage on the bond strength of self-etch adhesives bonded to dentin. Zander-Grande C, Loguercio AD, Stanislawczuk R, Martins GC, Gomes OM, Reis A. Am J Dent. 2011 Aug;24(4):239-44.
34. Durability of enamel bonding using one-step self-etch systems on ground and unground enamel. Reis A, Moura K, Pellizzaro A, Dal-Bianco K, de Andrade AM, Loguercio AD. Oper Dent. 2009 Mar-Apr;34(2):181-91.
35. Shear Bond Strength of Different MDP-Containing Adhesive Systems on Enamel and Dentin from Primary Teeth. Kim MJ, Kim J, Song JS, Chung SH, Hyun HK. J Clin Pediatr Dent. 2021 Jul 1;45(3):186-192.
36. A Comparative Evaluation of Shear Bond Strength of Embrace WetbondTM Pit and Fissure Sealant with or without the Use of a Universal Dental Adhesive System [ScotchbondTM Universal Adhesive] Using Different Bonding Protocols: A Multiparametric In Vitro Study. Swathy RS, Naidu J. Int J Clin Pediatr Dent. 2023 Jan-Feb;16(1):48-53.
37. Effect of different adhesive systems on dental defects and sensitivity to teeth in composite resin restoration: a systematic review and meta-analysis. Fang K, Chen K, Shi M, Wang L. Clin Oral Investig. 2023 Apr 5.
38. Comparison of Different Universal Adhesive Systems on Dentin Bond Strength. Brkanović S, Sever EK, Vukelja J, Ivica A, Miletić I, Krmek SJ. Materials (Basel). 2023 Feb 12;16(4):1530.
39. Defying ageing: An expectation for dentine bonding with universal adhesives? Zhang ZY, Tian FC, Niu LN, Ochala K, Chen C, Fu BP, Wang XY, Pashley DH, Tay FR. J Dent. 2016 Feb;45:43-52.