



Microshear bond strength of MDP-Containing Versus MDP-Free Bonding Systems: An in Vitro Study

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ABSTRACT

Aim: To evaluate the microshear bond strength of MDP-containing versus MDP-free bonding systems. **Materials and Methods:** 48 dentin rods were obtained from extracted teeth. Which were divided into two equal groups according to the type of bonding system (G): MDP free bonding system (G1), MDP containing bonding system (G2) (n=24). Each group was further subdivided into three subgroups according to storage time (T): immediate (T1), 3 months (T2), and 6 months (T3). The micro-bonding strength is tested by a universal testing machine. The results were statistically evaluated using the Shapiro-Wilk test, Levene's test, and post-hoc tests. **Results:** For specimens tested (T1), (G1) showed a non-significant lower mean shear bond strength value (12.18 ± 4.42) than (G2) (13.80 ± 4.61). (p-value=0.49). (T2), (G1) showed a non-significant higher mean shear bond strength value (10.79 ± 2.98) than (G2) (10.74 ± 4.59). (p-value=0.98). (T3), (G1) showed significantly higher mean shear bond strength values (8.61 ± 3.16) than (G2) (2.46 ± 0.95). (p-value =0.001). **Conclusions:** The adhesive procedure has a more effective impact on the final bond strength performance of restorations than the incorporation of MDP molecules.

Keywords: MDP containing bond system, MDP free bond system, Microshear bond strength.

1. Introduction

Achieving proper biological, mechanical, and esthetic qualities requires a durable bond between restorations and tooth structures. Effective bonding to the tooth structures is also necessary to prevent marginal leakage, caries adjacent to restoration, and pulp injury [1].

Thermal fluctuation, pH variation, and other aspects of the oral environment can affect bond durability [2]. Water absorption harms bond strength over time. Water molecules form hydrogen bonds with the polymer's polar regions, causing plasticization, expansion, and a subsequent decline in the polymer's mechanical properties [3].

Collagen fibril and adhesive resin hydrophilic component declination are thought to be the primary reasons for hybrid layer breakdown, which are broken down by two types of proteolytic enzymes: cathepsins (CTs) and metalloproteinases (MMPs) [4].

10-Methacryloxydecyl Dihydrogen Phosphate (MDP) exhibits the benefits of acid-base resistance and is believed to have a significant role in sealing restoration margins, preventing recurring caries defects, and enhancing restoration longevity. Both hydrophilic and hydrophobic moieties are present in functional monomers. Adhesive functional monomers promote chemical interaction and monomer penetration. Strong ionic bonds may be formed between MDP and calcium to create a salt with comparatively poor solubility. Several laboratory and clinical tests have demonstrated the regular adhesive action of adhesives containing MDP, particularly concerning long-term bond durability [5]. Functional monomers can provide stronger, more resistant, and durable bonds between adhesive and tooth structures as well as between resin composite and adhesive systems [6]. Testing for microshear bond strength (μ SBS) has significant clinical effects since the bond is subjected to different stresses, such as μ SBS and μ TBS [1,7]. Therefore, the current in vitro study was directed to compare μ SBS of MDP-containing versus MDP-free adhesive systems. The current study's null hypothesis was that the microshear bond strength would improve if MDP was added to the bonding system.

2. Materials and Methods

2.1. Materials:

MDP Free bonding system (BeautiBond light cure self-etching) (G1), MDP containing bonding system, (BISCO ALL-BOND UNIVERSAL Light Cured Dental Adhesive) (G2). And a nanohybrid resin composite (IVOCLAR VIVADENT TETRIC N CERAM A2). The materials used, their manufacturers, descriptions, and main parts were

included in Table 1.

2.2. Methods:

2.2.1. Sample size calculation:

Ensure enough power for a statistical test of groups with regard. Prior studies' findings were used to determine the effect size (f) of 0.803, the alpha (α) level of 0.05 (5%), and the beta (β) level of 0.05 (i.e., power=95%) [5]. A total of 48 samples, or 24 samples in each group, was the anticipated sample size (n).

2.2.2. Selection of teeth:

This study involved twelve newly removed caries-free adult permanent molars. The teeth were extracted for periodontal reasons. Immediately after extraction, scaled by both hand and ultrasonic scaler to remove calculus and remnants of periodontal tissues and examined by magnifying loops. Any tooth with caries, microcracks, or any other defects was eliminated. Before being used, teeth were kept at room temperature in distilled water.

2.2.3. Grouping of the specimens:

The teeth were utilized to acquire forty-eight (48) dentin rods. The rods were divided into two equal groups according to type of bonding system (G): MDP-free bonding system (G1), MDP-containing bonding system (G2) (n=24). Each group was further subdivided into three equal subgroups based on storage time (T): immediate (T1), 3 months (T2), and 6 months (T3).

2.2.4. Preparation of the specimens:

Acrylic resin blocks were made using a cylindrical Teflon mold, which included two opposing screws on top of a matched metal ring. During the acrylic resin setting process, the tooth was secured in place by the screws in a concentrated location parallel to the mold's long axis. Every sectioning approach in our investigation was conducted using Isomet 4000, a product from Buehler Ltd., located in Lake Bluff, Illinois, USA. In order to remove superficial

enamel and dentin beneath the occlusal surfaces were leveled off 2 mm beneath the superficial layer. The lubricant: water ratio was 1:30. An additional part was cut 2 mm cervical to dentino enamel junction.

2.2.5. Assessment of light intensity:

Light curing in this study was done by (Phantom wireless curing light, China)

2.2.6 Application of the restorative materials:

2.2.6.1. Application of MDP free bond system (G1):

Two separate coats of adhesive were applied using a bond brush. Each coat was rubbed for 10 seconds, excess solvent was removed using an air syringe for 3 seconds to achieve a uniformly glossy surface, then light cured for 5 seconds according to the manufacturer's instructions.

2.2.6.2. Application of MDP containing bond system (G2):

Two separate coats of adhesive were applied using a bond brush. Each coat was rubbed for 10 seconds, excess solvent was removed using an air syringe for 10 seconds to create a uniformly glossy surface, then light cured for 10 seconds according to the manufacturer's instructions.

2.2.6.3. Application of resin composite:

Application of a composite in a Tygon tube. Composite applicator and condenser Tygon tubes IVOCLAR VIVADENT TETRIC N CERAM resin composite, which was then cured for 10 seconds continuously by Phantom wireless curing light with wavelength 470 nm.

Four composite cylinders were placed. Each disk was submerged in distilled water for a full day. After a day, the tubes were cut open with a knife. Distilled water was refreshed daily [3].

2.2.7. Microshear bond strength testing:

Each tube was subjected to a μ SBS test using a 0.14-inch diameter stainless steel wire that was attached to the top movable head of the testing

apparatus and placed as near to the composite dentin contact as feasible. The shear bond strength in MPa was determined by dividing the force necessary for failure (Newton) by the surface area (mm^2).

2.2.8. Statistical analysis:

The data's normality was confirmed using the Shapiro-Wilk test. A two-way mixed ANOVA was used to investigate the effect of bond type on comparative shear stress at maximum load.

3. Results

3.1. Effect of bonding system on microshear bond strength:

For specimens tested immediately (T1), the MDP free bond system (G1) showed a lower mean shear bond strength value (12.18 ± 4.42) than the MDP containing bond system (G2) (13.80 ± 4.61). However, the two groups did not significantly differ from one another ($p\text{-value}=0.49$). After three months (T2), the MDP free bond system (G1) showed a higher mean shear bond strength value (10.79 ± 2.98) than the MDP containing bond system (G2) (10.74 ± 4.59). However, the two groups did not significantly differ from one another ($p\text{-value}=0.98$). After six months (T3), the MDP free bond system (G1) showed a higher mean shear bond strength value (8.61 ± 3.16) than the MDP containing bond system (G2) (2.46 ± 0.95). The two groups differed significantly from one another ($p\text{-value}=0.001$). (Table 2) and (Figure 1).

3.2. Effect of time on micro shear bond strength:

3.2.1 MDP free bonding system (G1):

Specimens tested immediately (T1) showed a higher mean shear bond strength value (12.18 ± 4.42) than specimens tested after three months (T2) (10.79 ± 2.98). However, the two groups did not significantly differ from one another ($p\text{-value}=1$). Specimens tested immediately (T1) showed a higher mean shear bond strength value (12.18 ± 4.42) than specimens tested after six months (T3)

(8.61 ± 3.16). However, the two groups did not significantly differ from one another (p-value=0.24). After three months (T2) showed higher mean shear bond strength value (10.79 ± 2.98) than specimens tested after six months (T3) (8.61 ± 3.16). However, the two groups did not significantly differ from one another (p-value=0.56). (Table 3) and (Figure 2).

3.2.2 MDP containing bond system (G2):

Specimens tested immediately (T1) showed a higher mean shear-bond strength value (13.80 ± 4.61) than specimens tested after three months (T2)

(10.74 ± 4.59). However, the two groups did not significantly differ from one another (p-value=0.34). Specimens tested immediately (T1) showed a higher mean shear-bond strength value (13.80 ± 4.61) than specimens tested after six months (T3) (2.46 ± 0.95). The two groups differed significantly from one another (p-value=0.001). Specimens tested after three months (T2) showed a higher mean shear bond strength value (10.74 ± 4.59) than specimens tested after six months (T3) (2.46 ± 0.95). The two groups differed significantly from one another (p-value=0.001). (Table 4) and (Figure 3).

Table 1: Materials' specification, composition, manufacturer, and lot number.

Materials	Specification	Composition	Manufacturer	LOT No.
BeautiBond Universal Adhesive	MDP-Free light-cured universal dental adhesive	<ul style="list-style-type: none"> Acetone Carboxylic acid monomer Phosphonic acid monomer HEMA free Bis-GMA (10 – 15 wt %) TEGDMA 6-methacryloyloxyhexyl phosphonoacetate (6-MHPA) 	https://www.shofu.de/en/	122012
BISCO ALL-BOND UNIVERSAL Light Cured Dental Adhesive	MDP-containing light cured universal dental adhesive	<ul style="list-style-type: none"> Ethanol solution (ethyl alcohol solution) 10 MDP (5 - 10 wt.%) 2-HEMA (10 – 30 wt %) Ethyl 4dimethylaminobenate (1 – 5 wt %) BisGMA (30 – 50 wt %) 	https://www.Bisco, Inc.	UN1170
IVOCAR VIVADENT TETRIC N CERAM (A2).	light-curing, radiopaque nano-hybrid resin composite.	<ul style="list-style-type: none"> dimethacrylates (19-20 wt.%). Fillers (80-81 wt. %). 	https://www.ivoclar.com/en_li	V17591

Table 2: Comparison of shear stress at maximum load between group 1 and group 2.

Shear strength (MPa)	MDP free bond system (G1)	MDP containing bond system (G2)	p-value $p < 0.05$	Significant
	Mean \pm SD	Mean \pm SD		
Immediate (T1)	12.18 \pm 4.42	13.80 \pm 4.61	0.49	NS
3 months (T2)	10.79 \pm 2.98	10.74 \pm 4.59	0.98	NS
6 months (T3)	8.61 \pm 3.16	2.46 \pm 0.95	0.001	S

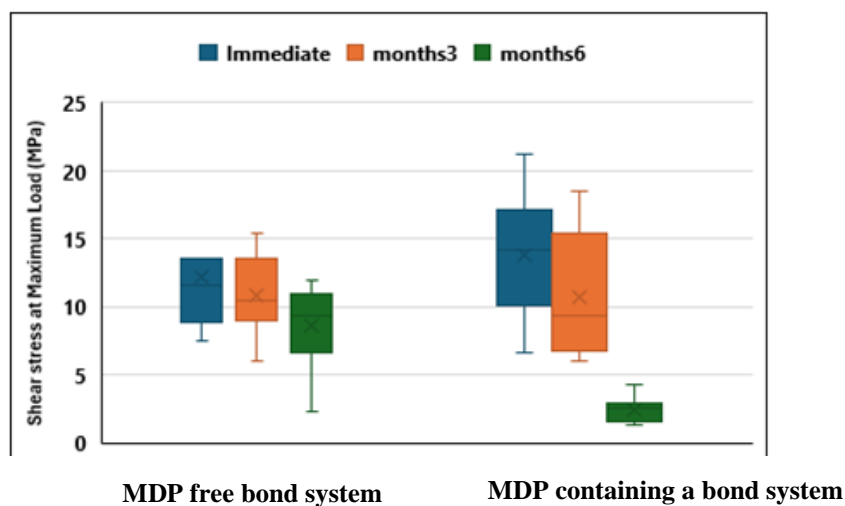


Figure 1: Boxplot for shear stress at maximum load of group 1 and group 2 at different times.

Table 3: Comparison of shear stress at maximum load between immediate (T1), 3 months (T2) and 6 months (T3) of MDP free bond system (G1).

Shear strength at maximum load (N)			
Mean ± SD			
Immediate (T1)	3 months (T2)		6 months (T3)
12.18 ± 4.42	10.79 ± 2.98		8.61 ± 3.16
Multiple comparison			
	MD	p- value p < 0.05	Significance
Immediate vs 3 months	1.39	1	NS
Immediate vs 6 months	3.57	0.24	NS
3 months vs 6 months	2.18	0.56	NS

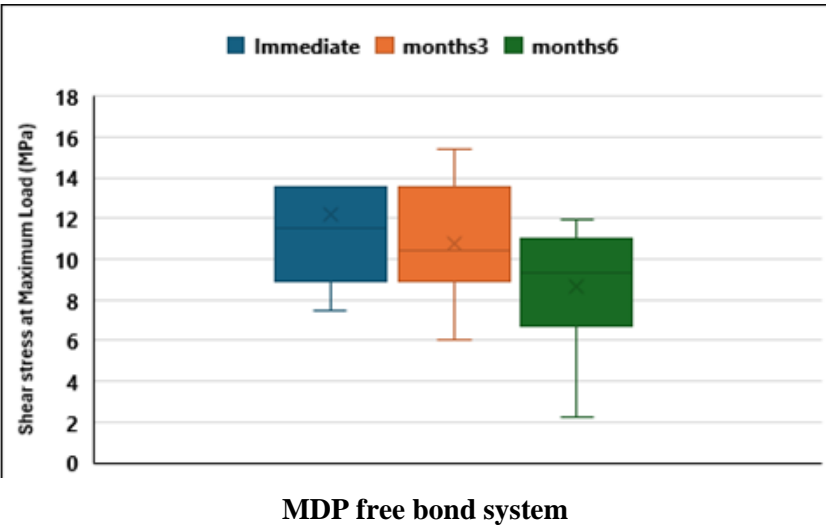


Figure 2: Boxplot for shear stress at maximum load of MDP free bonding system at different times (G1).

Table 4: Comparison of shear stress at maximum load between immediate (T1), 3 months (T2), and 6 months (T3) of MDP containing bond system (G2).

Shear strength at maximum load (MPa)			
Mean ± SD			
Immediate (T1)	3 months (T2)	6 months (T3)	
13.80 ± 4.61	10.74 ± 4.59	2.46 ± 0.95	
Multiple comparison			
	MD	p- value p < 0.05	Significance
Immediate vs 3 months	3.06	0.34	NS
Immediate vs 6 months	11.34	0.001	S
3 months vs 6 months	8.28	0.001	S

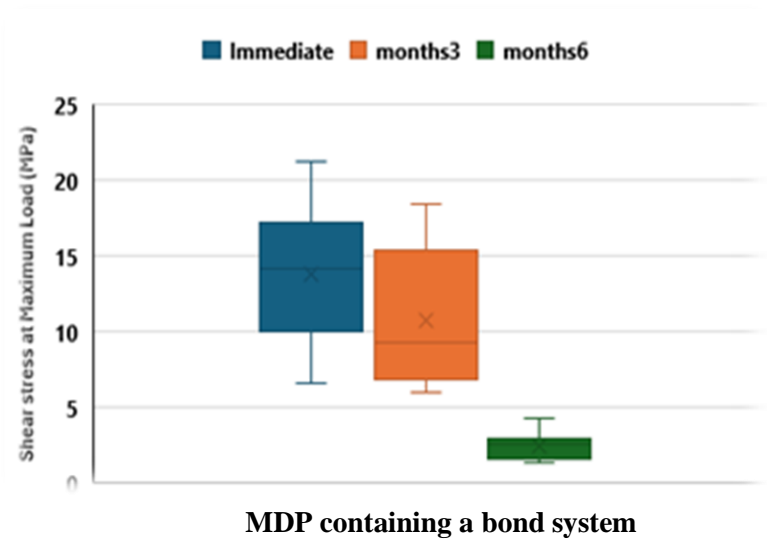


Figure 3: Boxplot for shear stress at maximum load of MDP containing bonding system at different times (G2).

4. Discussion

Dental biomaterials and tooth structure must form a durable bond. The development of 10-MDP primers, which provide reliable bonding solutions to enamel and dentin. Improving adhesive strength and durability is demonstrated by the way that 10-MDP and dental substrates interact chemically [1,9,10]. Therefore, this study evaluated the microshear bond strength of MDP-free versus MDP-containing bonding systems.

When comparing shear bond to tensile bond strength test, the shear bond strength test is simpler; this is the primary reason for its widespread use. An altered technique assessing the bonding ability of dentin-adhesive systems is the μ SBS test. The microshear bond strength test is superior to the macroshear bond strength test because it uses smaller specimens, which results in many specimens being able to be attached to the same dentin substrate because of the small size of the examined specimen. Additionally, many specimens were able to be bonded to the same dentin substrate due to the tested specimen's small size. The μ SBS test is crucial for clinical purposes because it can evaluate small portions of the tooth. In contrast with microtensile tests, which may cause early microcracking in the specimen, the test uses different substrates and prepares multiple specimens from the same tooth without the need for sectioning or trimming procedures [7,10,11].

The manufacturer claims that during a procedure of 30 seconds, a durable bond can be achieved. Its HEMA-free composition and the addition of a new polymerization catalyst may show promising bond strengths. So, it was considered to be compared to BISCO ALL-BOND UNIVERSAL Adhesive, which is an example of adhesives containing 10-MDP.

Adhesives used in this study were applied using the self-etch mode because etched dentin with 37.5% phosphoric acid had a markedly negative outcome. Numerous factors, such as excessive etching, insufficient phosphoric acid removal, insufficient

adhesive penetration to the denuded collagen network, and the removal of residual hydroxyapatite from the collagen mesh, which may jeopardize the possibility of adhesion, can contribute to the negative effects of phosphoric acid in dentin [13].

Adhesives used in this study were applied actively because both immediate and aged bond strength are increased by using the rubbing approach. By enhanced solvent evaporation and easier adhesive penetration into dentin tubule branches, using universal adhesives actively increased the bond of dentin. In actuality, the rate of monomer impregnation within the smear layer is increased when adhesives are actively applied employing a scrubbing approach. Consequently, the adhesive's quality interface is improved; therefore, there is less deterioration of the hybrid layer. [14].

Current adhesives have demonstrated strong bond strengths at 24 hours, but more investigation is always needed to determine how well they can seal dentinal tubules over time. Such adhesives cannot form an impermeable membrane that completely seals dentin, preventing water from passing through the tubules to the interface, so evaluations of microshear bond strength were done immediately and after 3 and 6 months [12].

The results of the study showed no significant difference between MDP-free bonding systems (BeautiBond Universal Adhesive) and MDP-containing (BISCO ALL-BOND UNIVERSAL Adhesive) and immediately and after three months. However, after six months, the MDP free bonding system showed a higher mean microshear bond strength value than the MDP containing bond system. Thus, the null hypothesis was rejected.

BISCO ALL-BOND UNIVERSAL contains 10-MDP demonstrates durable bond strengths to dentin, most likely because several inherent properties enable the development strong hybrid layer with dentin. 10-MDP's structure consists of a long hydrophobic 10-carbon chain, a methacrylate polymerizable end, and a short hydrophilic

functional phosphate component that can interact and ionize with hydroxyapatite. Bonding efficiency of SE adhesives is significantly influenced by the length of spacer chains of acidic functional monomers and their hydrophilicity. Collagen fibers are protected from degradation, it can generate. 10-MDP is a functional monomer that has been widely reported to be both safe and effective. Immediate dentin bond strength is increased when MDP is added to the adhesive resin [1,3,5,8,15].

Strengthening the bond immediately was enhanced by applying of ethanol and acetone wet bonding technique. Ethanol solvent in (BISCO ALL-BOND UNIVERSAL Adhesive) and acetone solvent in (BeautiBond Universal Adhesive). They are considered to be better than water as solvents because they can decrease collagen fibril width and increase interfibrillar space, which makes it easier for monomers to infiltrate collagen fibrils [14,15].

The strength of the bond decreased significantly over time (from 24 hours to six months). These results could be explained by the way water sorption gradually reduces bonding strength. Plasticization, swelling, and a resultant reduction in the mechanical properties of the polymer are caused by water molecules forming hydrogen bonds with the polar areas of the polymer. Additionally, storage of water and followed by sorption of water with time, which, as the adhesive matrix degrades and monomer auxiliary attachments are lost, also reduces the adhesives' bond strength. Furthermore, a gradual deterioration in bond strength may result from the fillers and matrix's bond failure [3,12,17].

BeautiBond Universal Adhesive, which contains carboxylic acid and polyacrylic acid, compared to 10-MDP, gives these monomers the capacity to combine with HAp to form Ca salts. Creating a reaction with dentinal apatite and a Ca-carboxylate salt, which results in the same bonding performance as the 10-MDP group. In addition to phosphonic/phosphoric acid's molecular

interactions with calcium, via the carboxylic acid component, the monomer mixture could possibly interact with the collagen fibrils in the substrate. Outside of the interfibrillar region, this interaction inhibits the transformation of (CaP) ions into apatite and enhances nucleation of hydroxyapatite. Therefore, dentin biomineralization due to the presence of (6-MHPA), a dual adhesive monomer included in BeautiBond, is claimed to be equally effective in bonding to enamel and dentine [12,18,19].

The findings of this study were in agreement with the results obtained by [8,11,12,17,19,20], who reported high SBS of BeautiBond Universal Adhesive and BISCO ALL-BOND UNIVERSAL Adhesive, (Abduljawad et al., 2024) explained the 10-MDP monomer's capability to form stable, water-insoluble MDP-Ca salts through adequate chemical interaction with hydroxyapatite, (Bacelar et al., 2017) explained There was no microleakage at the bonding area in the self-etch HEMA-free adhesive (BeautiBond), (Kim et al., 2021) explained how its functional monomer, 10-MDP, creates a very hydrophobic bond. By hindering a water-permeable adhesive layer of compromising bond performance, it may enhance bonding performance, (Morsy et al., 2020) outlined the chemical interactions between MDP and hydroxyapatite to create a stable nanolayer that may provide a stronger phase at the adhesive interface, improving the mechanical strength (Shakya et al., 2015). outlined 6-methacryloyloxyhexyl phosphonoacetate (6-MHPA), a dual adhesive monomer included in BeautiBond Universal Adhesive, is said to have the same bonding effectiveness as enamel and dentine and (Sultan et al., 2020) explained how the chemical bonding between calcium salts and 10-MDP could enhance its stability during storage and contribute to its slow rate of dissolution. However, [3,15,21], who reported different results, where HEMA in BISCO ALL-BOND UNIVERSAL Adhesive showed a decrease in bond strength,

(Costa et al., 2024) explained how HEMA may cause the adhesive layer to retain water, which could compromise mechanical strength. (Ghajari et al., 2019) explained that adhesive containing 10-MDP may contribute to water sorption and the gradual deterioration of bonding strength. (Wendlinger et al., 2023) outlined how the interaction between HEMA and MDP causes a considerable reduction in MDP chemical bonding and increases the susceptibility of water sorption adhesives containing HEMA to hydrolytic degradation, which results in significant degradation of the adhesive interface in the oral cavity.

5. Conclusions

- a. The adhesive procedure has a more effective impact on the final bond strength performance of restorations than the incorporation of MDP molecules.
- b. Gradual degradation of the bond between restoration and tooth, advocates using innovative methods to protect the interfacial zone.
- c. Further clinical studies are recommended to confirm the durability of the investigated bonding system.

Conflict of Interest

All authors declare that they have no conflict of interest.

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Ethics approval

The proposal of this study was approved by the Research Ethics Committee, Faculty of Dentistry, October 6 University. (Approval No. RECO6U/35-2023-November 6th, 2023)

Consent to Participate

Not applicable

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