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ORIGINAL RESEARCH

Evaluation of Microtensile Bond Strength in Indirect Composite Restorations to Deep Dentin using Two Different Bonding Systems at Different Decoupling Times

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Abstract

Background: Composite resins are widely used in restorative dentistry, yet polymerization shrinkage remains a significant challenge. This study aimed to evaluate the microtensile bond strength of indirect composite restorations to deep dentin with two different adhesive systems, considering varying decoupling times.

Materials and Methods: This experimental study involved 72 extracted molar teeth divided into six groups. Groups 1, 2, and 3 used Optibond FL, while groups 4, 5, and 6 used SE Bond. In groups 1 and 4, the bonding agent was applied, followed by decoupling with time (DWT) and a 1 mm-thick flowable composite. Groups 2 and 5 had a 0.5 mm composite applied, allowed a 5-minute DWT, then added another 0.5 mm composite. Groups 3 and 6 involved bonding and curing with a 1 mm composite but no DWT. All samples were restored with ENA Temp Micerium, and composite blocks were cemented. Micro-tensile bond strength was measured by applying a 500 N force until beam failure. Data were analyzed using ANOVA and Tukey's test (α = 0.05).

Results: Both Optibond FL and SE Bond adhesives, in combination with DWT, significantly increased the micro-tensile bond strength. Application of DWT at both stages (post-bonding and after the 0.5 mm flowable composite) resulted in higher micro-tensile bond strength compared to the groups without DWT.

Conclusion: The use of DWT significantly enhances the micro-tensile bond strength, and the timing of decoupling does not significantly affect the bond strength. Additionally, no significant differences were observed between the two types of bonding systems used.

Keywords: Composite Resins; Dental Bonding; Dentin; Tensile Strength; Dental Cements; Dental Restoration, Permanent; Analysis of Variance

Introduction

The reduction in tooth strength following extensive carious lesions and the associated increase in the risk of tooth fracture remain significant challenges in restorative dentistry (1). In the application of composite resins, particular attention must be paid to the polymerization process. Factors such as the elastic properties of the restorative material, the intensity of light irradiation, and the specific curing techniques used in both direct and indirect composite restorations can significantly influence the marginal adaptation and overall durability of the restoration (2, 3).

Most complications related to composite restorations are either directly or indirectly caused by polymerization shrinkage. During polymerization, a large number of monomer units link to form a polymer network, resulting in volumetric contraction that can exceed 5% in the final composite structure (4). The stress generated by this shrinkage is transmitted to the interface between the cavity walls and the composite resin, leading to adverse clinical

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outcomes. These may include cuspal deflection, the formation of craze lines, marginal discoloration, adhesive failure, interfacial gaps, and microleakage. Such complications can promote recurrent caries beneath the restoration and contribute to postoperative sensitivity, ultimately increasing the likelihood of restoration failure (5, 6).

Indirect composite restorations differ from direct techniques in terms of their impact on tooth reinforcement. Compared to direct restorations, indirect composites offer several advantages, including improved marginal sealing, primarily due to reduced polymerization shrinkage, enhanced occlusal and proximal morphology, increased resistance to wear, and superior biological compatibility as a result of a higher degree of polymerization (7). Nonetheless, a critical factor in restorative procedures is the long-term clinical performance and effectiveness of these restorations (8)

In the Delayed Dentin Sealing (DDS) technique, the pressure applied during the cementation of the final restoration can cause the collapse of demineralized dentin, particularly the exposed collagen fibers. This collapse compromises the hybrid layer formation and ultimately reduces the bond strength between the resin cement and dentin (9, 10). In contrast, the Immediate Dentin Sealing (IDS) technique has emerged as a widely adopted clinical approach in which a dentin bonding agent is applied immediately after tooth preparation and prior to final cementation (11, 12). The primary and most significant advantage of IDS lies in its ability to preserve the pulp–dentin complex and reduce postoperative sensitivity and bacterial microleakage during the provisional phase of treatment (13)

Immediate Dentin Sealing (IDS) can be performed using two distinct approaches. In the first method, following tooth preparation, only a dentin bonding agent is applied. In the second method, in addition to the bonding agent, a flowable composite is also placed over the adhesive layer (14). The incorporation of flowable composite offers several advantages, including the prevention of interference between the adhesive layer and impression materials during the impression-taking phase (15). Moreover, the flowable composite contributes to increased bond strength by preserving the integrity of the adhesive layer, absorbing polymerization stresses, and enhancing the long-term stability of the adhesive interface. IDS, when performed by applying a thin layer of resin bonding agent after preparation and allowing sufficient time for hybrid layer maturation (a process known as “decoupling with time”), has been shown to significantly improve the bond strength to dentin (16)

While some studies have reported no significant change or even an increase in the resin cement bond strength to dentin following the IDS technique (17, 18), other investigations have indicated a reduction in bond strength under similar conditions (19, 20). These contradictory findings highlight the need for further research to optimize adhesive protocols. Consequently, both researchers and clinicians are encouraged to explore and refine bonding techniques and materials that maximize tooth structure preservation, enhance patient comfort, and improve the long-term success of indirect bonded restorations.

The objective of the present study was to evaluate the microtensile bond strength of indirect composite restorations to deep dentin using two types of adhesive systems applied at different decoupling intervals

Materials and methods

In this experimental study, 72 sound molar teeth were extracted from patients aged 18 to 35 years who visited the clinical dentistry department at Azad University of Isfahan. All teeth were free from decay, cracks, and previous failures, in accordance with ethical guidelines. Third molars with severe anomalies or smaller than normal size, as well as samples that failed during the cutting procedure or slipped during microtensile testing, were excluded from the study.

Following extraction, the teeth were cleaned, and their roots were sectioned 2 mm below the cemento-enamel junction (CEJ) using a low-speed cutting machine (Cutting Machine Dental, Krupp Dental, 759DRZ, Hilzingen, Germany) under continuous water irrigation. The pulp chambers were then irrigated with a 2.5% sodium hypochlorite solution for cleaning.

To access deep dentin, the coronal portions of the specimens were divided into three equal segments and sectioned using a cutting machine (Cutting Machine Dentarapid, Krupp Dental, 759DRZ, Hilzingen, Germany) under water irrigation, between the cervical third and the occlusal surface, approximately 0.5 mm above the pulp. To standardize the smear layer, the exposed dentin surfaces were polished with 600-grit silicon carbide paper and mounted for further processing.

The bonding agents used in this study were Optibond FL (Kerr, USA) and SE Bond (Kuraray, Japan) (Figure 1). The samples were randomly divided into six experimental groups

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Figure1. Preparation and initial cutting of samples

Optibond FL is a fourth-generation adhesive system (etch-and-rinse, 3-step). In the first step, a 32% phosphoric acid was applied to the enamel and dentin for 15 seconds (starting with enamel and ending with dentin), then rinsed for 15 seconds and dried for 3 seconds. The second step involved applying the primer for 15 seconds, followed by air-drying for 5 seconds. The final step consisted of applying the bonding agent for 15 seconds, air-drying for 3 seconds, and curing for 20 seconds.

SE Bond is a sixth-generation adhesive system (two-step self-etching). This system was used in its self-etching mode. Initially, the primer was applied for 20 seconds and dried with gentle air. Subsequently, the bonding agent was applied, rubbed into the surface, followed by gentle air-drying, and cured for 10 seconds. Notably, the bonding layer in this system was applied in two layers.

In Group 1, Optibond FL was applied and cured using a light-curing device (Ultradent Cordless VALO LED Curing Light, USA). Following this, a dynamic water treatment (DWT) was performed for 5 minutes, and a 1-mm thick layer of flowable composite (Dentsply SDR Plus Bulk Fill Syringe Refill, Universal Shade, USA) was applied according to the manufacturer's recommendation.

In Group 2, Optibond FL (Kerr, USA) was applied and cured, followed by the application of 0.5 mm thick flowable composite (Dentsply SDR Plus Bulk Fill Syringe Refill, Universal Shade). After curing, DWT was performed for 5 minutes, and an additional 0.5 mm thick layer of flowable composite was applied.

In Group 3, Optibond FL was applied and cured, after which a 1-mm thick layer of flowable composite was placed, and no DWT was performed between stages.

In Group 4, SE Bond was applied and cured, followed by DWT for 5 minutes. Subsequently, a 1-mm thick layer of flowable composite was applied.

In Group 5, SE Bond was cured, then 0.5 mm thick flowable composite was applied. After curing, DWT was performed for 5 minutes, followed by the application of another 0.5 mm thick layer of flowable composite.

In Group 6, SE Bond was applied and cured, followed by the placement of a 1-mm thick flowable composite layer. No decoupling was performed between the stages.

Construction and cementing of resin component blocks:

Permanent restorations were fabricated for all samples using universal nanohybrid resin composite blocks (Filtek Z350, shade A1, 3M ESPE, USA) in silicone molds with a diameter of 12 mm and a height of 4 mm. After the removal of the temporary materials, the samples were cleaned by underwater flow for one week, followed by air-abrasive treatment using Al_2O_3 (Air Abrasion Master DN-NEO LX, China) for 10 seconds. Subsequently, a 32% phosphoric acid solution was applied to the surface, and adhesive bonding (All Bond Universal, Bisco, USA) was then applied.

The rationale for using this bonding agent was its compatibility with the resin cement used (Duo-Link, Bisco, USA). After distributing the resin cement (Duo-Link, Bisco, USA) onto the blocks, a cementation unit was employed to maintain a constant pressure of 1 kg for 5 minutes during the cementation process. Excess cement was removed using a microbrush. Curing was performed for 40 seconds from each of the buccal, lingual, and occlusal directions. The margins were carefully polished using Diacomp Plus Twist (EVE, Germany), and glycerin gel was applied and cured for an additional 20 seconds (Figure 2)

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Figure2. cementation of composite resin on the samples

The samples were stored in water for a period of 3 months (20). Each sample was then sectioned perpendicularly to the dental adhesive interface into beams with a surface area of 1 mm² using a CNC Cutting Section Machine (3 Axes Fully Automatic, Nemo Fanavaran Pars, Mashhad, Iran). Four samples with a surface area of 1 square millimeter were obtained from the central portion of the dentin of each tooth. The samples were fixed with cyanoacrylate glue to prepare for the microtensile bond strength test.

Subsequently, a tensile force of 500 N was applied at a speed of 0.5 mm/min until the beams fractured. The microtensile bond strength values of the samples were recorded using computer software connected to the Microtensile Machine MTD500plus (SDMechatronik, Feldkirchen Westerham, Germany) (Figure 3) (21, 22)

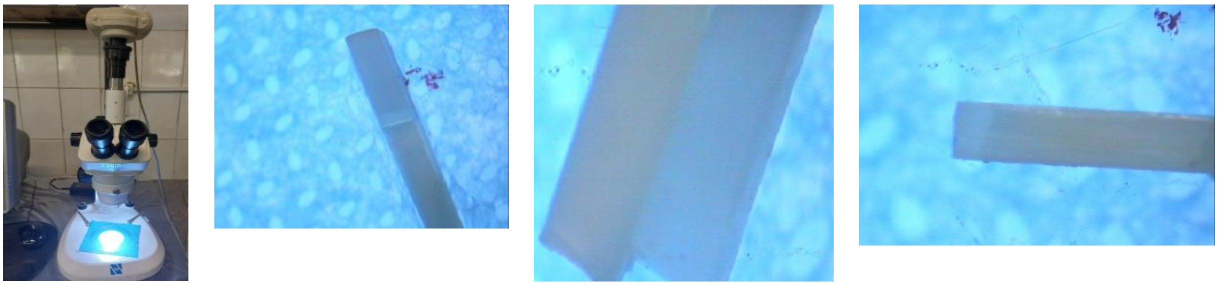

Figure3. Samples under the microscope

Fracture Analysis:

The fractured surfaces were examined under ×40 magnification using a Trinocular Zoom Stereo Microscope (SMP-200, HP, USA), equipped with a Moticam 480 Digital Camera (SP10.0224, Motic Instruments Inc, CA, USA), to determine the type of fracture. These fractures were classified into the following categories (20):

1. Cohesive in dentin (CD)
2. Adhesive between cement and dentin (AD)
3. Adhesive between indirect restoration and resin cement (ADR)
4. Cohesive in composite resin (CR)
5. Hybrid or mixed

The normality of the data was assessed using the Shapiro-Wilk test. Data were analyzed using independent analysis of variance (ANOVA) and Tukey's HSD post-hoc test, utilizing SPSS version 26 software. 5% was considered significant.

Results

Based on descriptive statistics, the highest mean microtensile bond strength corresponds to group one, which is Optibond FL with DWT after bonding and before using flowable composite, and the lowest mean microtensile band strength corresponds to the sixth group, which is the SE control group without DWT (Figure 4).

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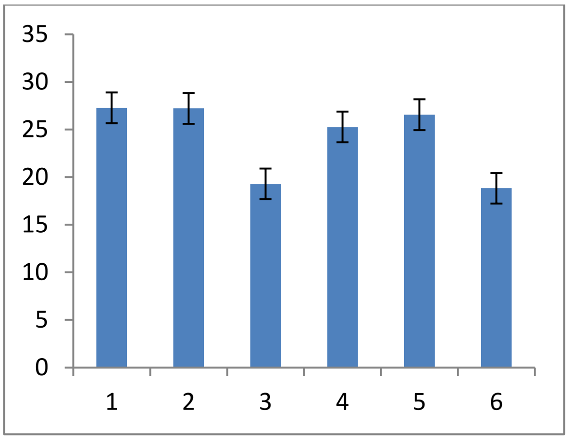

Figure 4. The mean microtensile bond strength of bonded samples by bonding type

According to the analysis of variance (ANOVA), a significant difference was observed between the mean micro tensile bond strength values across the different bonding types (P < 0.001).

Based on the Tukey HSD post-hoc test, the results presented in Table 1 indicate that the mean microtensile bond strength of Optibond FL bonded samples with DWT after bonding (Group 1) was significantly greater than the microtensile bond strength of the Optibond FL control group without DWT (Group 3) and the SE control group without DWT (Group 6) (P < 0.001).

Additionally, the mean microtensile bond strength of the Optibond FL bonded samples with DWT after the composite thin layer (Group 2) was significantly greater than the microtensile bond strength of both the Optibond FL control group without DWT (Group 3) (P < 0.001) and the SE control group without DWT (Group 6) (P < 0.001).

The mean microtensile bond strength of the Optibond FL control group without DWT (Group 3) was significantly lower than that of the SE group with DWT after bonding (Group 5) (P < 0.001) and the SE bonded samples with DWT after bonding (Group 4) (P < 0.001).

Moreover, the mean microtensile bond strength of the SE bonded samples with DWT after bonding (Group 4) was significantly greater than the microtensile bond strength of the SE control group without DWT (Group 6) (P < 0.001).

The average microtensile bond strength of the SE bonded samples with DWT after the composite thin layer (Group 5) was also significantly greater than the microtensile bond strength of the SE control group without DWT (Group 6).

No statistically significant differences were observed among the other groups. In other words, regardless of the type of bonding or whether the DWT groups were evaluated after bonding or after the application of the thin composite layer, no significant differences were identified among them. However, a significant difference was noted when comparing the DWT groups to those without DWT, with the microtensile bond strength being significantly higher in the DWT groups.

The type of bonding used and the sequence of DWT application (before or after the composite layer) did not result in significant differences between the groups, as determined by ANOVA. The only factor that caused a significant difference between the groups was whether the DWT process was performed

Table 1. Pair-by-pair comparison of the mean microtensile bond strength of bonded samples in the studied groups

Groups	1	2	3	4	5	6
1	-	NS	S	NS	NS	S
2	NS	-	S	NS	NS	S
3	S	S	-	S	S	NS
4	NS	NS	S	-	NS	S
5	NS	NS	S	NS	-	S
6	S	S	NS	S	S	-

S: The mean difference is significant at the 5% level
NS: The mean difference is not significant at the 5% level

According to the chi-square test results, no significant difference was observed in the location of the fracture based on the type of bonding (p = 0.871). In other words, the fracture location of indirect composite restorations bonded to deep dentin using either Optibond FL or SE Bond, after the application of bonding and a 5-minute decoupling time, was not significantly different. Similarly, no significant difference was found after using bonding and flowable composite with a thickness of 0.5 mm, followed by a 5-minute decoupling time (Table 2).

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Table 2. Frequency and percentage of composite fracture location based on bonding type

Groups	AD	MI	CR	ADR	CD	P value
	N (%)	N (%)	N (%)	N (%)	N (%)	
1	9 (17.3)	2 (25)	1(16.7)	0(0)	0(0)	0.871
2	10(19.2)	1 (12.5)	1(16.7)	0(0)	0(0)	
3	8(15.4)	2(25)	0(0)	2(33.3)	0(0)	
4	7 (13.5)	2 (25)	1(16.7)	2(33.3)	0(0)	
5	9 (17.3)	1 (12.5)	1(16.7)	1(16.7)	0(0)	
6	9 (17.3)	0(0)	2(33.3)	1(16.7)	0(0)	

AD: Adhesive between cement and dentin
MI: mix
CR: Cohesive in composite resin
ADR: Adhesive between indirect restoration and resin cement
CD: cohesive in dentin

Discussion

According to the results of the present study, the use of both types of bonding agents (Optibond FL and SE Bond) in conjunction with Dentin Wetting Technique (DWT) at both stages—after bonding and after the application of a 0.5 mm composite layer resulted in a significant increase in micro-tensile bond strength compared to the control group without DWT. Notably, no significant difference was observed between the two bonding types. Additionally, the timing of decoupling (whether it occurred immediately after bonding or following the application of the 0.5 mm composite layer) did not influence the micro-tensile bond strength. In other words, the decoupling time, whether preceding or following the composite application, did not impact the micro-tensile bond strength.

In a review by Harden et al. (23), it was demonstrated that the Immediate Dentin Sealing (IDS) technique enhances bond strength, irrespective of the adhesive system used. The subgroup analysis indicated that the use of a three-step etch-and-rinse adhesive system or the combination of an adhesive system with a flowable composite layer provided better bond strength, which is consistent with the findings of the present study. Considering the critical role of the hybrid layer in the restoration, it is important to note that this layer, in the 5th and 7th generations of bonding agents, is not sufficiently hydrophobic, which can lead to increased permeability of dentin. This permeability, in turn, can result in marginal microleakage and porosity within the hybrid layer. Furthermore, the movement of water between the layers, coupled with the water tree effect, contributes to the degradation of the interface. For this reason, fourth- and sixth-generation bonding agents were utilized in this study.

Compared to Delayed Dentin Sealing (DDS), IDS has been shown to significantly increase both micro-tensile bond strength (μTBS) and shear bond strength (SBS) (24). Moreover, the use of the IDS technique demonstrates significant advantages over DDS, as supported by previous research (25).

In the study by Gailani et al. (26), the bond strength of universal bonding agents was found to be dependent on the type of material used. Optibond FL, which has high filler content and superior mechanical resistance, exhibited the highest bond strength values. The bond strength was influenced by the components of each bonding agent, varying between the two different approaches—Immediate Dentin Sealing (IDS) and Delayed Dentin Sealing (DDS). Understanding the specific components in each bonding system is crucial for optimizing bond strength.

In the study by Ferreira-Filho et al. (20), all groups in which IDS was employed showed higher bond strength values compared to the control group. This can be attributed to the superior demineralization capacity and better hybrid layer formation of etch-and-rinse and self-etch adhesives, compared to self-adhesive resin cements used in the control group. When immediate dentin sealing and resin coating are allowed to establish a bond within the first 5 to 30 minutes, the bond durability is significantly improved, even under occlusal loading conditions (16).

In a systematic review by Varadan et al., the bonding performance of indirect restorations using an enhanced IDS technique (involving bonding and flowable composite) was compared to conventional IDS (bonding without flowable composite). The study concluded that enhanced IDS exhibited equal or superior bond strength compared to conventional IDS strategies. The addition of a low-viscosity resin

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composite layer creates a thicker bond layer, prevents the repositioning of dentin during the final restoration, facilitates better preparation in less office time, and eliminates potential undercuts. Therefore, enhanced IDS offers superior preservation of dentin integrity compared to conventional IDS methods (17).

Overall, IDS enhances bond strength, reduces cracks, minimizes bacterial leakage, and alleviates dentin sensitivity. The use of a dentin bonding agent with filler, or the combination of a dentin bonding agent without filler with a flowable composite, enhances both the clinical and technical aspects of IDS (11).

In the present study, a flowable composite was applied over the bonding layer. When bonding agents with lower filler content are used, the flowable composite not only supports the bonding layer but also plays a significant role in the restoration process. This may reduce the thickness of the IDS layer during sandblasting. IDS, when combined with low-viscosity composite, increases the thickness of the hybrid layer and alleviates some of the stress generated by the shrinkage of the resin cement during polymerization. Additionally, the low-viscosity composite absorbs a portion of the stress introduced during the application of force in the micro-tensile bond strength test. Consequently, less force is applied to the bonded surfaces. The increase in the hybrid layer thickness during IDS with dentin bonding agent (DBA) and low-viscosity composite has been shown to enhance bond strength (27).

Polymerization shrinkage stress is directly related to the amount of shrinkage and the elastic modulus of the material. Specifically, the more elastic the composite, the better it can reduce contraction stress (28). In the study by Alleman et al. (16), immediate dentin sealing with a dentin bonding system, followed by the application of a thin resin layer and allowing time for the hybrid layer to develop (between 5 and 30 minutes), significantly improved bond strength. Thus, the decoupling over time helps prevent the stress caused by contraction, particularly when larger volumes of material are used.

Decreasing the speed of the polymerization reaction allows adequate time for the material to flow, thereby reducing the stress induced by polymerization shrinkage. Decoupling with time (DWT) provides sufficient duration for the dentin bonding system to develop a mature hybrid layer, effectively resisting any shrinkage stresses associated with subsequent composite layers bonded to it (16). Furthermore, DWT is able to alleviate the differences between various types of dental hard tissues involved in bonding methods (29).

In the present study, a decoupling time of 5 minutes was used. The time required for the dentin hybrid layer to mature can range from 5 minutes for shallow direct composite restorations to 2 weeks for bonded indirect final restorations (16), which aligns with the findings of other studies (30, 31). The primary objective of restorative dentistry protocols is to apply small volumes of composite that can move towards the developing hybrid layer of dentin during treatment. This beneficial dynamic movement is possible only if the initial volume of composite placed over the dentin bonding system remains thin (less than 1.5 mm) during the first 5 minutes of the polymerization reaction (32). In other words, composite layers thicker than 1 mm fail to integrate with the developing hybrid layer, at least during the first 5 minutes of polymerization. Thin layers move towards the tooth, thereby enhancing the strength of the hybrid layer, whereas thicker layers move away from the tooth, towards the center of mass, potentially weakening or breaking the hybrid layer (33).

The lack of a significant difference between the two stages of decoupling (after the bond and after using the composite thin layer) in the Optibond FL group may be attributed to the unique properties of this adhesive. Optibond FL contains fillers such as fumed SiO2, barium, aluminoborosilicate, and NaSiF6, which impart radiopacity and enable the formation of a uniform adhesive layer approximately 88 microns in thickness. This feature diminishes the need for additional protection from flowable composite (34). Similarly, SE bond also includes micro-fillers that enhance the adhesive layer's thickness and function as a cushion during polymerization, thereby partially neutralizing polymerization shrinkage stresses. Additionally, bonding agents with fillers exhibit reduced shrinkage, which enhances the seal between the restorative material and the substrate (35).

In the present study, the mean bond strength in groups that used SE bond with DWT performed after the application of the 0.5 mm composite layer was higher than that observed in groups where DWT was performed immediately after bonding. This suggests that when bonding agents with lower filler content are used, it is preferable to support the bonding layer with a thin composite layer before performing DWT. Moreover, these bonds typically employ mild acids,

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achieving deep dentin demineralization (up to one micron). This process leaves behind mineral agents like hydroxyapatite, which remain in place and interact with collagen, thus creating an optimal porosity for micromechanical retention. These adhesives also form a chemical bond (MDP) between hydroxyapatite and functional monomers at the molecular level, thereby enhancing resistance to microleakage (36).

Regarding failure analysis, the present study revealed that indirect restorations using both Optibond FL and SE bonds, after a 5-minute decoupling time, either following bonding or after applying a 0.5 mm flowable composite layer, showed no significant failure. Most failures occurred at the adhesive-dentin interface (AD), characterized by adhesive failure, regardless of whether dentin was filled or not. This finding aligns with Ferrina's study (20), which reported that the majority of failures occurred at the AD interface. Similarly, Van den Breemer's study found the highest failure rate at the dentin-adhesive interface, while Galliani's study reported that the highest incidence of failure occurred at the adhesive-restoration interface, with the lowest occurring at the dentin-adhesive interface.

Conclusion

The results of the present study showed that the use of both types of bonding agents in combination with Decoupling with Time (DWT) significantly enhances the micro-tensile bond strength, with no significant differences between the two bonding agents. The application of DWT, regardless of the timing (post-bonding or after the placement of a 0.5 mm composite layer), consistently improves the micro-tensile bond strength when compared to groups without DWT. This finding suggests that DWT is an effective technique for enhancing the bond strength in adhesive dentistry.

Conflict of Interests: The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial, or non-financial in this article

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