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Impact of immediate versus delayed dentin sealing on adhesive luting effectiveness

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ABSTRACT

Objective: To evaluate the effect of immediate versus delayed dentin sealing (IDS/DDS) on the micro-tensile bond strength (μTBS) of CAD/CAM composite adhesively luted onto dentin.

Methods: Upon sandblasting, silanization and adhesive application, CAD/CAM composite slabs were luted (AL) onto dentin using composite following eight protocols: $_1$**'DDS$_{LC}$'**: C-SE2 light-cured (LC) on dentin prior to AL; $_2$**'DDS$_{prov}$'**: temporary, saliva storage, C-SE2 LC, AL; $_3$**'DDS$_{noLC}$'**: C-SE2 (non-LC), AL; $_4$**'DDS$_{pulpless_noLC}$'**: 'DDS$_{noLC}$' with the pulp chamber filled with composite (non-vital tooth); $_5$**'IDS$_{ctrl}$'**: C-SE2 LC, flowable composite, sandblasting, etching, C-SE2 (non-LC), AL; $_6$**'IDS$_{sal}$'**: 'IDS$_{ctrl}$' but saliva storage before AL; $_7$**'IDS$_{wat}$'**: 'IDS$_{non_contaminated}$' but water storage before AL; $_8$**'IDS$_{prov}$'**: 'IDS$_{ctrl}$' but IDS covered with temporary, saliva storage before AL. After one-week water immersion, half of the specimens were cut for immediate μTBS testing and the other half submitted to 1,200,000 chewing cycles, cut into μ-specimens and submitted to 10,000 thermal cycles prior to μTBS testing. Linear mixed-effects statistics were applied ($\alpha = 0.05$).

Results: All four IDS and the two DDS approaches that involved separate adhesive light-curing, resulted in reliable immediate and aging-resistant bonding effectiveness to dentin. Using DDS, all μ-specimens failed prior to testing when the adhesive was not separately light-cured prior to luting onto vital dentin. When luted onto non-vital dentin, significantly higher μTBS was recorded that nevertheless remained significantly below that measured for the four IDS and two DDS approaches.

Conclusion: No difference in adhesive luting effectiveness was recorded between IDS and DDS when the adhesive was separately light-cured at the dentin side prior to luting.

1. Introduction

The challenge of achieving durable long-term bonding to dentin using resin monomers has driven significant advancements in dental adhesive technology over the years [1,2]. Despite these advancements, bonding to enamel remains more reliable than bonding to dentin because of the unique nature of the latter complex tissue [1–4]. Teeth in need of semi-direct or indirect restorations often exhibit extensive damage and tooth-structure loss, resulting in a substantial amount of exposed dentin. Since the success of CAD/CAM partial restorations depends highly on bonding of the restorative material onto tooth structure, achieving a strong, aging-resistant bond to dentin is crucial for prolonged clinical success [5–8]. It has been reported that the optimal time to bond to dentin is immediately after finishing tooth preparation, as this minimizes contamination from bacteria, provisional cement, or impression material [4,5,8].

In the 1990s, the concepts of "dual bonding" and "resin coating" were first described. The "dual bonding" technique involves applying an adhesive onto dentin immediately after tooth preparation, whereas the "resin coating" technique prescribes the additional application of a low-viscosity composite on top of the adhesive, which is applied to both dentin and enamel [9–14]. These techniques aim to reduce dentin

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permeability, prevent contamination, minimize pulpal irritation following preparation, and increase bond strength [5,9,11–13]. Later, the concept of sealing dentin immediately after tooth preparation became known as "immediate dentin sealing" (IDS) [5,15]. The key difference between IDS and the "resin coating" technique is that IDS focuses solely on sealing freshly cut dentin [5,11,13]. Additionally, IDS introduced the use of a filled adhesive to seal freshly cut dentin, suggesting an alternative to using an unfilled bonding agent followed by a low-viscosity composite layer [5,15].

Numerous studies, including clinical trials, meta-analyses and systematic reviews, have assessed the benefits of IDS compared to the conventional delayed dentin sealing (DDS) technique prior to adhesively luting an indirect restoration [4,8,16–27]. These studies together highlight numerous advantages of IDS over DDS, including (1) reduced dentin permeability, which protects the dentin/pulp complex, decreases postoperative sensitivity and also minimizing the need for a provisional, (2) prevention of improper bonding due to contamination with blood, saliva, and temporary cement remnants, and (3) enhanced resistance of the adhesive interface to the polymerization shrinkage stress imposed during cementation.

Despite extensive research over the years, the impact of IDS on the bonding performance of semi-direct or indirect restorations remains inconclusive. Systematic reviews and meta-analyses reveal a lack of randomized clinical trials (RCTs) and long-term *in-vitro* studies comparing the benefits of IDS to DDS [8,16,21,23,24,26]. For example, a recent systematic review by Ozer F et al. [8] included 60 studies, of which only five evaluated IDS over the long term. Another recent systematic review and meta-analysis by Alghauli MA et al. [4] on the clinical benefits of IDS, included six RCTs and five non-RCTs. Their findings suggest benefits of IDS over DDS but recommend further long-term RCTs to confirm their conclusions.

While RCTs provide higher levels of evidence than *in-vitro* studies, they cannot determine the precise causes of failure because restorations in the oral cavity are subjected to various simultaneous stresses [28]. On the other hand, *in-vitro* studies are easier to conduct compared to RCTs and typically precede RCTs in dental research [28,29]. *In-vitro* studies can subject the specimens to extensive aging to test the durability of the bonding interface under stress [30]. Therefore, the aim of this *in-vitro* study was to evaluate the effect of IDS on the micro-tensile bond strength (μTBS) to dentin, hereby comparing eight different adhesive luting protocols for semi-direct and indirect composite/ceramic restorations. The study groups were designed to evaluate the effect of IDS versus DDS in various simulated clinical scenarios, such as pulpless teeth, single-appointment semi-direct restorations, provisional restorations preceding adhesive luting, and IDS having been exposed to saliva during the interim period before final restoration placement. Specimens also underwent extensive aging to stress the bonding interface. The null hypotheses tested were that light-curing the adhesive together with the composite cement (1), tooth vitality (2), IDS (3), provisionalization (4), and aging (5) would not affect μTBS to dentin.

2. Materials and methods

2.1. Specimen preparation

Resin-composite CAD/CAM blocks (Katana Avencia 14 L, shade A2 LT, Kuraray Noritake, Tokyo, Japan) were sectioned into three slabs (14.5 × 14.5 × 5.0 mm) using a semi-automatic precision cutting machine (Accutom-50, Struers, Ballerup, Denmark) under water irrigation. The intaglio surface was ground with a humidified #120-grit diamond grinding disc (Galaxy Diamond Disc Red, QATM, Mammelzen, Germany) for 10 sec using a grinder/polisher machine to simulate the surface characteristics of CAD/CAM milling [31].

One hundred and twenty-eight non-carious human third molars were collected following informed consent approved by the Commission for Medical Ethics of KU Leuven (file number S64350). All teeth were stored in distilled water at 4°C, used within six months after extraction, and randomly allocated into sixteen experimental groups (n = 16). Teeth were mounted in gypsum blocks with the tooth crown exposed, upon which the occlusal thirds were removed using a slow-speed diamond saw (Micracut 151, Metkon, Bursa, Turkey). After manually removing the enamel ring of each tooth using a high-speed medium-grit (107 μm) diamond bur (806.314.142.524.014, Meisinger, Neuss, Germany), a uniform smear layer was produced on all dentin surfaces using a high-speed fine-grit (46 μm) diamond bur (8881.314.014, Komet Dental, Lemgo, Germany). A new diamond bur was used for each experimental group. The exposed dentin surfaces were examined for absence of enamel and exposure of pulp tissue using a stereomicroscope (Stemi 2000-CS, Zeiss, Oberkochen, Germany). After smear-layer preparation and surface inspection, teeth were stored at 37°C in 100 % relative humidity for at least 30 min. The materials used in this study are presented in Table 1.

2.2. Resin-composite CAD/CAM slab intaglio surface treatment

The intaglio surface of the composite CAD/CAM slabs was marked with a permanent marking pen (Sharpie, Sanford L.P., Oak Brook, IL, USA), and sandblasted until the mark was completely eliminated using a sandblasting device (RONDOflex plus 360, Kavo, Birberach, Germany) loaded with 29-μm aluminum oxide (Velopex, London, UK) at 2 bar (0.2 MPa) pressure, positioned at 1 cm and 90° to the surface, with continuous small circular movements and moving the nozzle tip from side to side. Upon sandblasting, the CAD/CAM slabs were ultrasonically cleaned in acetone for 2 min to remove residual sand particles, followed by thorough drying with an oil-free air syringe. Afterwards, the intaglio surface of the CAD/CAM slabs was etched with phosphoric acid (K-Etchant Syringe, Kuraray Noritake) for 5 sec, thoroughly rinsed with distilled water, and air-dried with an oil-free air syringe. Silane (Clearfil Ceramic Primer Plus, Kuraray Noritake) was applied, left undisturbed for 60 sec to allow the solvent to evaporate, and then air-dried with an oil-free air syringe. A layer of Clearfil SE Bond 2 'bond' (Kuraray Noritake) was next applied on the intaglio surface of the CAD/CAM slab, air-thinned but not light-cured, upon which the pre-treated slab was covered from light until being adhesively luted.

2.3. Fabrication of 3D-printed custom-made temporary restorations

A square prism measuring 10 × 10 × 2 mm was first designed using FreeCAD software (FreeCAD 0.19.4, FPA, Anderlecht, Belgium). The .STL file was imported into a slicing software (Composer 1.3.3, Asiga, Alexandria, Australia), where supports were added, slices were created with a 50-μm thickness, and the 3D printer's (Pico 2 HD, Asiga) settings were adjusted according to the manufacturer's instructions for the 3D-printable light-curing composite used (Temp PRINT, GC, Tokyo, Japan). Before starting the printing process, the 3D-printable composite was shaken for approximately 2 min before being poured into the 3D-printer reservoir.

Upon printing completion, the printed restorations were removed from the printing platform and placed into a glass with isopropanol solution (>96 %), which, in turn, was placed in an ultrasonic water bath for 2 min. The restorations were then removed, air-dried, and the cleaning procedure was repeated in a clean isopropanol solution (>96 %) in an ultrasonic water bath for another 2 min. After air-drying, final polymerization was carried out by curing for 3 min in a post-curing unit (Asiga Flash, Asiga) on the side opposing the supports side. After removing the supports, the other side was post-cured for additional 3 min.

The side opposite of the supports side had a flat surface, which was selected as the bonding surface for the temporary restorations. This surface was sandblasted using the sandblasting device (RONDOflex plus 360, Kavo) loaded with 29-μm aluminum oxide (Velopex) at 2 bar (0.2 MPa) pressure. Sandblasting was done with the nozzle positioned

Table 1
Materials (in alphabetical order) used in the study, presenting their composition, application protocol and batch numbers(s).

Materials	Composition	Application protocol	Batch no.
Aluminum oxide 29-µm (Velopex)	Aluminum oxide, titanium dioxide	Sandblast the surface using a 2 bar (0.2 MPa) pressure, with the nozzle tip positioned at 1 cm and 90° to the surface, with continuous small circular movements, moving the nozzle tip from side to side	240322
Clearfil AP-X (Kuraray Noritake)	Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, accelerators, pigments (shade A3.5)	1. Apply the resin composite on the intaglio surface of pre-treated CAD/CAM composite block 2. After a constant weight of 1 kg over the course of 1 min, light-cure each surface for 20 sec.	A60040
Clearfil Ceramic Primer Plus (Kuraray Noritake)	Ethanol, γ-MPTS, 10-MDP	1. After pre-treating the intaglio surface of the CAD/CAM composite slab, dispense the necessary amount of Clearfil Ceramic Primer Plus into a well of the mixing dish immediately before application. 2. Apply Clearfil Ceramic Primer Plus to the intaglio surface of the CAD/CAM composite slab with an applicator brush. 3. After 60 sec, dry the entire intaglio surface sufficiently using mild, oil-free air flow.	BR0069
Clearfil SE Bond 2 (Kuraray Noritake)	**Primer:** 10-MDP, HEMA, hydrophilic aliphatic dimethacrylate, dl-camphorquinone, water **Bond:** 10-MDP, HEMA, Bis-GMA, hydrophobic aliphatic dimethacrylate, dl-camphorquinone, initiators, accelerators, silanated colloidal silica	1. Apply the primer in the tooth structure with an applicator brush in an active motion for 20 sec. 2. Dry sufficiently with mild air for more than 5 sec until the primer does not move. 3. Apply bond with an applicator brush. 4. Make a uniform bond film using a gentle airflow. 5. Light-cure for 10 sec.	000011
Clearfil Majesty Flow HIGH (Kuraray Noritake)	Triethylene glycol dimethacrylate, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica dl-camphorquinone, accelerators, pigments (shade A1)	1. Apply a small amount over the adhesive layer. 2. Light-cure for 10 sec.	C80027
GC Temp PRINT (GC)	UDMA, quartz, 2,2′-ethylenedioxydiethyl dimethacrylate, esterification products of 4,4′-isopropylidenediphenol, ethoxylated and 2-methylprop−2-enoic acid, diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide, 2-(2H-benzotriazol−2-yl)-p-cresol (shade medium)	1. Create slicing data with a layer thickness of 50 µm and send it to the additive manufacturing device (Pico 2 HD, Asiga, Alexandria, Australia). 2. Select the proper print program for GC Temp PRINT including all relevant process parameters. 3. Before pouring, shake GC Temp PRINT in the original bottle for approximately 2 min. 4. After shaking, directly pour it into the reservoir and set both the reservoir and the platform in accordance with the additive manufacturing system instructions. 5. Start printing. 6. Carefully remove the printed restorations from the printing platform. Do not remove the support structures under the objects. 7. For cleaning, place the restorations in a glass container with an isopropanol solution (>96 %), then place the glass in an ultrasonic water bath for 2 min. 8. Dry parts with compressed air. 9. Repeat the cleaning procedure in a fresh isopropanol solution (>96 %), then place in an ultrasonic water bath for 2 min. 10. After cleaning, check if the surface is still shiny, wet or sticky, which would indicate there is still isopropanol or residual monomers on the printed object. If such, please repeat the cleaning procedure until restorations	1910101

(*continued on next page*)

Table 1 (*continued*)

Materials	Composition	Application protocol	Batch no.
		are completely clean. 11. Proceed with the final polymerization, cure for 3 min on opposite from support side. 12. Remove supports, then apply light for another 3 min from the other side.	
Katana Avencia Block (Kuraray Noritake, Tokyo, Japan)	Mixed filler with colloidal silica and aluminum oxide, cured resins consisting of methacrylate monomer (copolymer of UDMA and other methacrylate monomers), pigments (shade A2 LT)	1. Roughen the intaglio surface by blasting with alumina powder (29 μm) using air pressure of 0.2 MPa. 2. Etch the intaglio surface with phosphoric acid gel for 15 sec, rinse well and dry. 3. Apply Clearfil Ceramic Primer Plus, leave it for 60 sec and dry.	000507
K-Etchant Syringe (Kuraray Noritake)	Phosphoric acid (25–45 %)	1. Etch for 10 sec, rinse thoroughly with water and dry.	AD0184
Oxyguard (Kuraray Noritake)	Glycerol, polyethyleneglycol, catalysts, accelerators, dyes	1. Apply a thin layer over the light-cured flowable composite. 2. Additionally light-cure for 20 sec. 3. Rinse-off with air-water spray.	1C0107
Temp-Bond NE (Kerr)	**Base:** Zinc oxide, white mineral oil (petroleum). **Catalyst:** Octanoic acid, 2-ethoxybenzoic acid, (R)-p-mentha−1,8-diene	1. Extrude equal lengths of base and accelerator onto a mixing pad. 2. Thoroughly mix the pastes for approximately 30 sec.	8155555

Bis-GMA: bisphenol A diglycidylmethacrylate; **TEGDMA:** triethyleneglycol dimethacrylate; **γ-MPTS:** γ-methacryloxypropyltrimethoxysilane; **10-MDP:** 10-methacryloyloxydecyl dihydrogenphosphate; **HEMA:** 2-hydroxyethyl methacrylate; **UDMA:** urethane dimethacrylate.

1 cm away and 90° to the surface, using continuous small circular movements until the entire surface became dull. Upon sandblasting, the temporary restorations were ultrasonically cleaned in acetone for 2 min to remove residual sand particles, followed by thorough drying with an oil-free air syringe.

2.4. Bonding protocol

The bonding protocol differed among the eight experimental groups in the current study, as detailed below and schematically summarized in Table 2:

1. **'DDS_{LC}'**: Clearfil SE Bond 2 ('C-SE2', Kuraray Noritake) 'primer' was actively rubbed on dentin for 20 sec, repeatedly having applied fresh primer liquid, and dried with mild air until no visible movement of the primer was observed anymore, while leaving a glossy surface. A C-SE2 'bond' layer was next applied, strongly air-thinned and light-cured for 10 sec using the LED light-curing unit (LCU) SmartLite Pro (Dentsply Sirona, Konstanz, Germany) with a light output of 1,250 mW/cm^2, as specified by the manufacturer and verified regularly during the project using a Marc Resin Calibrator (Bluelight Analytics, Bluelight Analytics, Halifax, NS, Canada). The restorative resin-based composite Clearfil AP-X ('C-APx'; Kuraray Noritake) was applied as luting agent on the intaglio surface of a pre-treated resin composite CAD/CAM slab, which was next positioned onto the dentin surface of the tooth. Luting was performed under a constant weight of 1 kg over the course of 1 min. Excess composite (cement) was removed with a burnisher (Ladmore #2, Hu-Friedy, Chicago, IL, USA) prior to light-curing for 20 sec from each side at a 1-mm distance (buccal, mesial, lingual, distal and occlusal; 100-sec total curing time).
2. **'DDS_{noLC}'**: Following the protocol described for $_1$'DDS_{LC}', with the exception that the C-SE2 'bond' layer was not strongly air-thinned and not light-cured before adhesively luting the composite CAD/CAM slab. In other words, the C-SE2 'bond' layer was light-cured together with the composite luting agent (C-APx).
3. **'DDS_{prov}'**: After tooth-surface preparation, a custom-made 3D-printed temporary (GC Temp PRINT, GC) was luted with a non-eugenol temporary cement (Temp-Bond NE, Kerr, Orange, CA, USA). The specimens were stored for two weeks in human saliva at 37°C, which was refreshed every other day. Upon removal of the provisional, any remaining non-eugenol temporary cement was removed using a sandblasting device (RONDOflex plus 360, Kavo) loaded with 29-μm aluminum oxide (Velopex) at 2 bar (0.2 MPa) pressure, positioned at 1 cm and 90° to the surface, with continuous small circular movements and moving the nozzle tip from side to side. The subsequent bonding and luting protocol followed exactly that described for $_1$'DDS_{LC}'.
4. **'$DDS_{pulpless\_noLC}$'**: Unlike the other groups, the pulp chamber of the teeth in this group was accessed through the root furcation, upon which pulpal tissue was removed with a dentin excavator, followed by roughening the internal pulp chamber with a high-speed medium-grit (107 μm) diamond bur (806.314.142.524.014, Meisinger) to further clean it. The chamber was then filled with C-APx (C-SE2 was before applied following manufacturer's instructions, with C-SE2 'primer' (Kuraray Noritake) additionally being actively rubbed onto the dentin surface for 20 sec and C-SE2 'bond' (Kuraray Noritake) gently air-thinned and light-cured for 10 sec). The teeth were next left in a flask at room temperature for 1 week to dehydrate. Then, they were embedded in gypsum blocks with the tooth crown exposed and stored in distilled water at 4°C for 2 weeks to rehydrate. The bonding procedure followed the protocol described for $_2$'DDS_{noLC}'.
5. **'IDS_{ctrl}'**: C-SE2 'primer' (Kuraray Noritake) was actively rubbed on dentin for 20 sec, repeatedly having applied fresh primer liquid, and dried with mild air for 10 sec until no movement of the primer was observed anymore, hereby leaving a glossy surface. A C-SE2 'bond' (Kuraray Noritake) layer was next applied and light-cured for 10 sec. A thin layer of flowable composite (Clearfil Majesty ES Flow, Kuraray Noritake) was applied over the bond layer and light-cured for 10 sec. Oxyguard (Kuraray Noritake) was applied over the flowable composite and additional light-curing took place for another 20 sec. The air barrier was rinsed-off and the tooth was air dried. Then, the resin-coated surface was sandblasted using a sandblasting device (RONDOflex plus 360, Kavo) loaded with 29-μm aluminum oxide (Velopex) at 2 bar (0.2 MPa) pressure, positioned at 1 cm and 90° to the surface, with continuous small circular movements and moving the nozzle tip from side to side until the entire surface appeared dull. Afterwards, the IDS surface was etched with 37 % phosphoric acid (K-Etchant Syringe, Kuraray Noritake) for 15 sec followed by thorough water rinsing for 10 sec and air-drying. C-SE2 'primer' and 'bond' (Kuraray Noritake) were next applied according to manufacturer's instructions, followed by gently air-thinning (no light-curing). Resin composite (C-APx; Kuraray Noritake) was applied on the intaglio surface of a pre-treated resin-composite CAD/CAM slab

Table 2
Experimental DDS and IDS protocols detailed step by step.

	DDS_{noLC}	$DDS_{pulpless\_noLC}$	DDS_{prov}	DDS_{LC}	IDS_{sal}	IDS_{wat}	IDS_{prov}	IDS_{ctrl}
1. Tooth de-/rehydration	no	yes	no	no	no	no	no	no
2. Dentin smear-layer preparation	yes	yes	yes	yes	yes	yes	yes	yes
3. C-SE2 application followed by light-curing	no	no	yes	yes	yes	yes	yes	yes
4. Flowable composite application	no	no	no	no	yes	yes	yes	yes
5. Provisional restoration	no	no	yes	no	no	no	yes	no
6. 2-week storage	no	no	saliva	no	saliva	distilled water	saliva	no
7. Particulate air-abrasion	no	no	yes, dentin	no	yes, IDS	yes, IDS	yes, IDS	yes, IDS
8. 37% phosphoric-acid application	no	no	no	no	yes, IDS	yes, IDS	yes, IDS	yes, IDS
9. Adhesive luting of restoration	yes	yes	yes	yes	yes	yes	yes	yes

and next positioned on the dentin surface of the tooth. Luting was performed under a constant weight of 1 kg over the course of 1 min. The excess luting agent was removed with a burnisher (Ladmore #2, Hu-Friedy) prior to light-curing for 20 sec from each side at a 1.0 mm distance (buccal, mesial, lingual, distal and occlusal; 100-sec total curing time).

6. **'IDS_{sal}'**: After IDS following the protocol described for $_5$'IDS_{ctrl}', the specimens were stored for two weeks in human saliva at 37°C, which was refreshed every other day. The bonding procedure followed the protocol described for $_5$'IDS_{ctrl}'.
7. **'IDS_{wat}'**: After IDS following the protocol described for $_5$'IDS_{ctrl}', the specimens were stored for two weeks in distilled water at 37°C, which was refreshed every other day. The bonding procedure followed the protocol described for $_5$'IDS_{ctrl}'.
8. **'IDS_{prov}'**: After IDS following the protocol described for $_5$'IDS_{ctrl}', a 3D-printed custom-made temporary was provisionally luted with a non-eugenol temporary cement (Temp-Bond NE, Kerr). Then, the specimens were stored for two weeks in human saliva at 37°C, which was refreshed every other day. The bonding procedure followed the protocol described for $_5$'IDS_{ctrl}'.

The human saliva used to store the specimens for the 'DDS_{prov}', 'IDS_{sal}', and 'IDS_{prov}' groups was collected from the same donor during the preparation of each respective study group.

2.5. Micro-tensile bond strength

After one week of storage in distilled water at 37°C, the teeth, except those in the '$DDS_{pulpless\_noLC}$' group, had their root sectioned using a low-speed diamond saw (Micracut 151, Metkon) under constant water irrigation. The pulp chamber was emptied with a dentin excavator, cleaned with a high-speed medium-grit (107 μm) diamond bur (806.314.142.524.014, Meisinger), and then filled with C-APx (C-SE2 was before applied following manufacturer's instructions, with C-SE2 'primer' (Kuraray Noritake) additionally being actively rubbed onto the dentin surface for 20 sec and C-SE2 'bond' (Kuraray Noritake) gently air-thinned and light-cured for 10 sec).

Next, half of the teeth of all groups (n = 64) were cut using a low-speed diamond saw (Accutom-50, Struers) into micro(μ)-specimens with 1.0 × 1.0-mm cross-sectional dimensions and 10-mm length for μTBS testing. The dimensions of the sticks were precisely measured using a digital caliper (CD-S15CK, Mitutoyo, Kawasaki, Japan), from which the cross-sectional area was calculated. The μ-specimens were then fixed to a μTBS testing jig using cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan) and tested in tension mode at a crosshead speed of 1.0 mm/min using an LRX testing machine (Lloyd, Hampshire, UK) equipped with a 100 N load cell. The bond strength was calculated in MPa by dividing the imposed force (in N) at the time of fracture by the bonded area (in mm^2).

The other half of the teeth (n = 64) were aged in a chewing simulator (Chewing Simulator – System Willytec, SD Mechatronik, Feldkirchen-Westerham, Germany), using a stainless steel antagonist sphere with 6-mm diameter, loaded on the occlusal plane for 1,200,000 cycles (1.6 Hz, 50 N). Upon chewing aging, the restored teeth were cut into μ-specimens with 1.0 × 1.0-mm cross-sectional dimensions and 10-mm length, and aged for 10,000 (5–55°C; with a 30 s dwell time) thermocycles (Thermocycler THE-1200, SD Mechatronik) to determine the 'aged' μTBS [32,33]. The number of μ-specimens tested in each experimental group is mentioned in Table 2.

The micro-tensile bond-strength test was conducted strictly following the Academy of Dental Materials guidelines [34].

2.6. Light microscopy (LM) and scanning electron microscopy (SEM) failure analysis

Upon μTBS testing, the failure mode of each μ-specimen was analyzed by observing the fractured two halves by LM (Stemi 2000-CS, Zeiss, Oberkochen, Germany), this to categorize the failure mode as either 'cohesive failure in dentin', 'cohesive failure in restoration', 'adhesive failure at the cement/dentin interface', 'cohesive failure within adhesive', 'adhesive failure at the cement/restoration interface', or 'mixed failure'. One μ-specimen representative of each experimental group with a μTBS close to the mean and one pre-testing failure, when available, were selected for SEM failure analysis. Specimens were fixed in 2.5 % glutaraldehyde in 0.1 M sodium cacodylate buffer, rinsed with 0.2 M sodium cacodylate buffer, gradually dehydrated in concentration-increasing ethanol solutions, and dried using hexamethyldisilazane (HMDS). Next, they were mounted onto stubs and gold-sputter coated (JFC-1300, JEOL, Tokyo, Japan) to be finally examined using SEM (JSM-6610LV, JEOL).

2.7. SEM of the adhesive interface morphology

One additional tooth of each dentin sealing protocol (DDS and IDS) was prepared following the 'DDS_{LC}' and 'IDS_{ctrl}' protocols described before. Upon preparation, the teeth were cut using a low-speed diamond saw (Accutom-50, Struers) into two halves and fixed in 2.5 % glutaraldehyde in 0.1 M sodium cacodylate buffer, rinsed with 0.2 M sodium cacodylate buffer, gradually dehydrated in concentration-increasing ethanol solutions and then dried using hexamethyldisilazane (HMDS).

The specimens were embedded in a self-cure epoxy resin (Epofix, Struers). After 24 h, the sample was ground using a grinder/polisher (Beta with Vector power head, Buehler, Lake Bluff, IL, USA) with wet silicon-carbide sandpaper to a specific size of 11 × 8 × 2 mm. The specimen was then mounted on a specific holder for cross-section polishing (CP). Before CP polishing, the sample underwent pre-polishing using a JEOL polishing holder and a custom-made device. This manual polishing was performed with 30-μm lapping film (3 M, Saint Paul, MN, USA) and distilled water. The interface surface was then cross-section polished using an argon-ion beam (SM-09010, JEOL) for 12 h. The accelerating voltage was set to 5.0 kV with an ion-current density of 25 mA/cm^2. Next, the specimens were gold-sputter coated (JFC-1300, JEOL) and finally examined using SEM (JSM-6610LV, JEOL, Tokyo, Japan) [35].

2.8. Micro(μ)-CT evaluation

A tracer solution of ammoniacal silver nitrate (pH ranging from 8.5 to 8.9) was freshly prepared by dissolving 50 g of silver-nitrate crystals in 25 ml of distilled water. Then, concentrated (28 %) ammonium hydroxide was added to the black solution until it turned clear, when the ammonium ions complexed the silver into diamminesilver(I) ions ([Ag$(NH_3)_2]^+$) [36]. Representative μ-specimens from 'DDS_{LC}', '$DDS_{pulpless\_noLC}$' and 'IDS_{ctrl}' were processed for μCT. As all 'DDS_{noLC}' μ-specimens failed during sectioning, this experimental group could not be included. The μ-specimens were immersed in the ammoniacal silver-nitrate solution for 12 h in the dark at room temperature. Afterwards, the excess silver nitrate was removed with distilled water and the μ-specimen was placed in a solution of photographic developer (Dextol, Kodak, Rochester, NY, USA) under fluorescent light for 8 h. The specimens were then scanned using a μCT scanner (UniTOM HR, Tescan, Brno - Kohoutovice, Czech Republic), with the μ-specimens placed into a customized holder immersed into distilled water. The scanning parameters were 80 kV and 100 μA, with an exposure time of 200 ms. The pixel size was 7.5 μm, with a rotation step of 0.4 degrees and an averaging of 3 frames. 3D-images were acquired through 3D-reconstruction, followed by 3D-modeling and 3D-analysis software (CTVOX, Bruker, Billerica, MA, USA). Cross-sectional images were obtained to analyze the interfaces of (1) IDS/DDS with dentin, (2) composite cement with IDS/DDS, and (3) composite cement with the CAD/CAM block.

2.9. Statistical analysis

Linear mixed-effects (LME) statistical modeling with specific contrasts (R-4.0.3; R Foundation for Statistical Computing, Vienna, Austria) [37] was conducted to compare the immediate and aged μTBS of the different experimental groups investigated, with differences considered statistically significant at $\alpha = 0.05$. The variables 'Bonding mode', 'Dentin sealing', and 'Aging' were fixed variables, while 'Tooth' was employed as the random variable.

3. Results

The overall μTBS data (immediate and aged) of the experimental groups are detailed in Table 2 and graphically presented in boxplots in Fig. 1. The statistical analysis of the LME model revealed that the main variable 'Bonding mode' and 'Aging' significantly contributed to the statistical model ($p < 0.0001$) (Table 3). The first-, and second-order

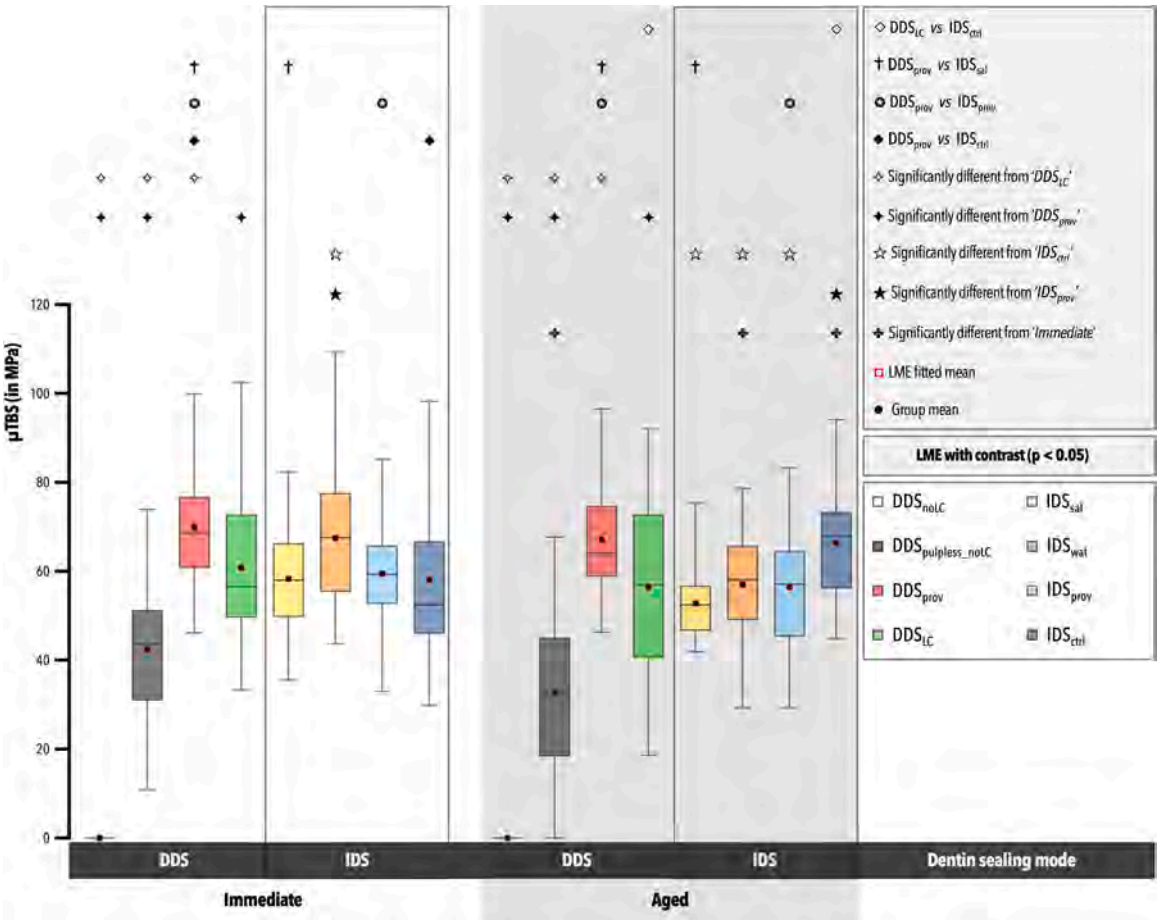

Fig. 1. Box-and-whisker plots of the 'immediate' and 'aged' μTBS recorded for the four DDS and four IDS experimental groups investigated. The LME statistical results are indicated with specific symbols explained in the plot legend.

Table 3
Mean micro-tensile bond strength (μTBS in MPa) and fitted LME means for the experimental DDS and IDS groups investigated.

Experimental groups	Immediate			Aged		
	Mean μTBS (SD[a])	PTF[b]/n[c]	Fitted LME mean	Mean μTBS (SD)	PTF/n	Fitted LME mean
DDS_{noLC}	0.0 (0.0)	64/64	0.0	0.0 (0.0)	64/64	0.0
$DDS_{pulpless\_noLC}$	42.3 (15.1)	0/64	42.3	32.6 (17.4)	3/61	32.6
DDS_{prov}	70.0 (12.3)	0/56	69.9	67.1 (12.2)	0/64	67.1
DDS_{LC}	60.9 (16.5)	0/64	60.9	56.4 (17.9)	0/64	56.4
IDS_{sal}	58.2 (11.0)	0/64	58.2	52.9 (7.6)	0/64	52.9
IDS_{wat}	67.5 (14.1)	0/64	67.5	57.0 (12.1)	0/64	57.0
IDS_{prov}	59.5 (9.3)	0/64	59.5	56.4 (12.6)	0/64	56.4
IDS_{ctrl}	58.1 (16.1)	0/64	58.1	66.4 (12.3)	0/64	66.4

[a] SD = standard deviation;
[b] PTF = pre-test failure;
[c] n = number of specimens.

interactions were analyzed, revealing a first-order interaction for the variables 'Bonding mode' and 'Aging' and a second-order interaction for 'Bonding mode x Aging', meaning that these variables individually or combined influenced the μTBS of the specimens tested.

While specific significant differences in μTBS were recorded among the experimental groups, overall, the four IDS approaches as well as the two DDS approaches that involved separate light-curing of the adhesive prior to actual luting, resulted in reliable immediate and aging-resistant bonding effectiveness to dentin.

Regarding immediate μTBS recorded following the DDS approach, the significantly highest μTBS was recorded for 'DDS_{prov}' (70.0 ± 12.3 MPa). Slightly but significantly lower μTBS was recorded for 'DDS_{LC}' (60.9 ± 16.5 MPa). No immediate μTBS was recorded when the adhesive was not separately light-cured prior to adhesive luting onto

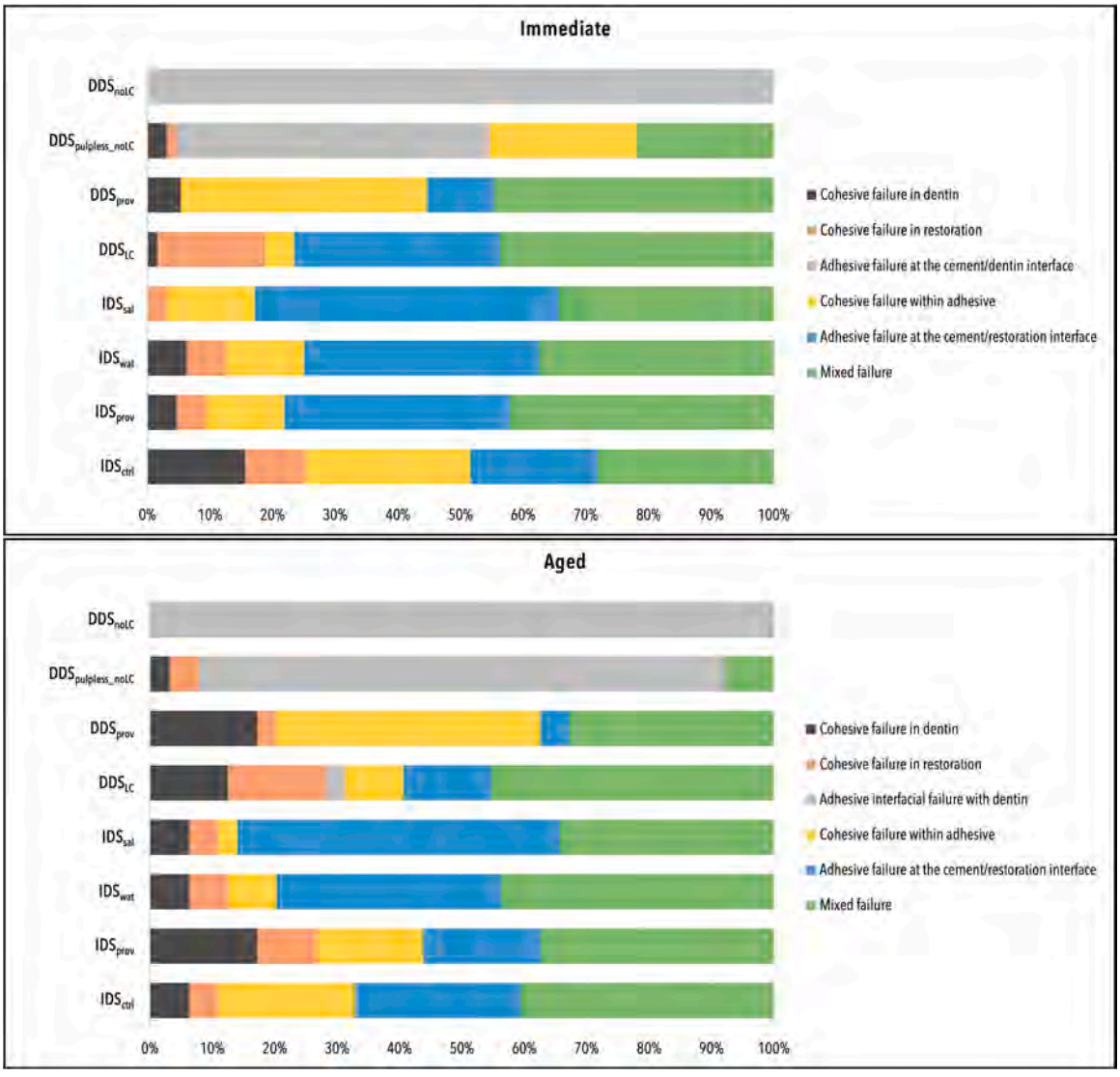

Fig. 2. 'Immediate' and 'aged' light-microscopy failure modes recorded for the eight DDS and IDS experimental protocols investigated in this study.

vital dentin ('DDS_{noLC}': all pre-testing failures). When adhesively luted onto non-vital dentin ('$DDS_{pulpless\_noLC}$'), much higher μTBS (42.3 ± 15.1 MPa) was recorded that nevertheless remained significantly below that recorded for 'DDS_{LC}' and 'DDS_{prov}'. Mechano-thermally aging the DDS specimens resulted in a similar μTBS outcome trend among the four DDS experimental groups as was recorded immediately. Aging did not significantly decrease μTBS, except for a slightly but significantly lower μTBS recorded for '$DDS_{pulpless\_noLC}$' (aged: 32.6 ± 17.4 MPa; immediate: 42.3 ± 15.1 MPa). Specimens that failed before actual testing (PTF) were explicitly counted as 0.0 MPa in further analysis and thus considered in the calculation of the μTBS means.

Regarding immediate μTBS recorded following the IDS approach, the significantly highest μTBS was recorded for 'IDS_{wat}' (67.5 ± 14.1 MPa), as compared to 'IDS_{ctrl}' (58.1 ± 16.1 MPa) and 'IDS_{prov}' (59.5 ± 9.3 MPa), with their immediate μTBS also be in line with that recorded for 'IDS_{sal}' (58.2 ± 11.0 MPa). Upon mechano-thermally aging the IDS specimens, the significantly highest μTBS was recorded for 'IDS_{ctrl}' (66.4 ± 12.3 MPa), with the aged μTBS recorded for 'IDS_{sal}' (52.9 ± 7.6 MPa) and 'IDS_{wat}' (57.0 ± 12.1 MPa) not being significantly lower than that of 'IDS_{prov}' (56.4 ± 12.6 MPa). Aging solely slightly but significantly reduced μTBS recorded for 'IDS_{wat}' (aged: 57.0 ± 12.1 MPa; immediate: 67.5 ± 14.1 MPa), while a slightly but significantly higher μTBS upon aging was recorded for 'IDS_{ctrl}' (aged: 66.4 ± 12.3 MPa; immediate: 58.1 ± 16.1 MPa).

Comparing the highest immediate DDS versus IDS μTBSs recorded, 'DDS_{prov}' (70.0 ± 12.3 MPa) versus 'IDS_{wat}' (67.5 ± 14.1 MPa), no significant difference was found ($p = 0.482$). Comparing the highest aged DDS versus IDS μTBS recorded, 'DDS_{prov}' (67.1 ± 12.2 MPa) versus 'IDS_{ctrl}' (66.4 ± 12.3 MPa), no significant difference was found ($p = 0.946$).

The fracture-mode distribution of all μ-specimens is presented in Fig. 2. All 'DDS_{noLC}' μ-specimens failed adhesively at the cement/dentin interface, when tested immediately or upon aging. While more different failure modes were recorded for '$DDS_{pulpless\_noLC}$', 'adhesive failure at the cement/dentin interface' remained the predominant failure mode, in particular recorded more upon aging. Most other experimental groups revealed more variable failure modes, with 'mixed failures' and 'adhesive failure at the cement/restoration interface' being recorded most frequently. 'DDS_{prov}' μ-specimens also presented with a significant percentage of 'cohesive failures within adhesive'. Aging in a chewing simulator for 1,200,000 cycles followed by 10,000 thermocycles did overall not have a notable impact on the failure-mode distribution. (Table 4)

Representative SEM images of fractured μ-specimen pairs are presented in Figs. 3 and 4, illustrating the failure modes of specimens exhibiting a μTBS close to the mean and representing each aged experimental group. In the case of 'DDS_{noLC}', the pre-testing failure (PTF) mode is illustrated when the specimen was tested immediately.

Argon-ion-beam cross-section polished interfaces of representative specimens are shown in Fig. 5, revealing interfacial porosities and cracks at both the adhesive-dentin and adhesive-cement interfaces for 'DDS_{noLC}'. The thickness of the adhesive-resin and composite-cement layers was, respectively, 15 and 48 μm on average. Tight multi-layer interfaces without interfacial porosities were imaged when the adhesive was separately light-cured, either following 'DDS_{LC}' or 'IDS_{ctrl}'. Strong air-drying of the adhesive resin, followed by separate light-curing, resulted in an about 10-μm film thickness covered by an about 35-μm composite-cement layer for 'DDS_{LC}'. 'IDS_{ctrl}' resulted in an about 15-μm adhesive-resin layer covered by an about 100-μm thick flowable-composite layer and about 120-μm thick composite-cement layer.

The μCT-scanned multi-layered interfaces of 'DDS_{ctrl}', '$DDS_{pulpless\_noLC}$' and 'IDS_{ctrl}' with dentin, exposed to silver-nitrate infiltration, are presented in Fig. 6. Nano-leakage indicated by the presence of silver particles was only observed for '$DDS_{pulpless\_noLC}$'.

Table 4
Linear mixed-effects (LME) statistical analysis for the 1st and 2nd order interactions.

	Df	denDF	F-value	p-value
Bonding mode	7	992	1.778.338	< .0001*
Aging	1	992	0.82186	< .0001*
Bonding mode x Aging	7	992	262.408	< .0001*

* Statistically significant.

4. Discussion

This study evaluated the effectiveness of various adhesive luting protocols for semi-direct and indirect restorations across eight simulated clinical scenarios. The primary objective was to compare the adhesive luting performance of the *Delayed Dentin Sealing* (DDS) and *Immediate Dentin Sealing* (IDS) approaches, with the latter being widely supported by multiple meta-analyses and systematic reviews [4,8,21,24,26,27]. A critical factor in the DDS approach is whether the adhesive should be light-cured separately before seating the restoration. While pre-curing may increase the cement space between the tooth substrate and the restoration, potentially compromising the fit, most manufacturers recommend curing the adhesive simultaneously with the luting composite after the restoration has been seated. Since water sorption from the dentin-pulp complex can interfere with adhesive luting, the DDS approach was also tested on non-vital (dry) dentin. In both DDS and IDS luting protocols, the impact of using an intermediate provisional restoration between tooth preparation and the final luting phase was investigated. Additionally, the effect of aging in water versus saliva on the IDS approach was analyzed.

The first bonding protocol evaluated in this study involved simultaneously light-curing the adhesive and the composite cement ('DDS_{noLC}'). However, all specimens using this protocol failed before testing (pre-testing failures or *PTFs*), leading to the rejection of the first hypothesis (1) that curing the adhesive together with the composite cement would not affect μTBS to dentin. This result underscores the necessity of separately light-curing the adhesive to ensure stable bonding to dentin when luting semi-direct or indirect restorations. These findings align with previous studies that tested various luting materials [10,11,38,39–41]. As noted in earlier research, delaying the light-curing of the adhesive allows the interface to absorb water from the underlying dentin [3,40,42–44]. This effect is particularly pronounced in cases involving highly permeable or deep dentin. Interfacial porosities were observed when the adhesive was not pre-cured but instead cured simultaneously with the composite cement. These irregularities compromised the adhesive interface, likely have contributed to PTFs.

In this respect, it is important to highlight that this study utilized third molars from young patients, which have highly permeable dentin. Such dentin likely exacerbated water sorption at the adhesive interface, representing a worst-case scenario compared to more typical clinical conditions where semi-direct or indirect restorations are bonded to less permeable, sclerotic, or even amalgam-corroded dentin. In such clinical conditions, interfacial water sorption is reduced, minimizing its impact on adhesive luting when the adhesive is co-cured with the composite cement.

Insufficient polymerization caused by light attenuation when simultaneously curing the adhesive and composite through the restoration is unlikely to have contributed to the PTFs. Although light intensity at the adhesive interface was reduced due to the interposed restoration, this effect was minimized by implementing an extended 100-second light-curing time and applying high-intensity LED irradiation (1,250 mW/cm^2) from five sides at a 1-mm distance. A recent *in-vitro* study by Hardy et al. [43] further supports this, demonstrating that sufficient polymerization can be achieved even with 6-mm-thick restorations made from the same composite CAD/CAM material (shade A3 LT) and bonded using the same adhesive and composite cement employed in the current study. Given that the simulated restorations in

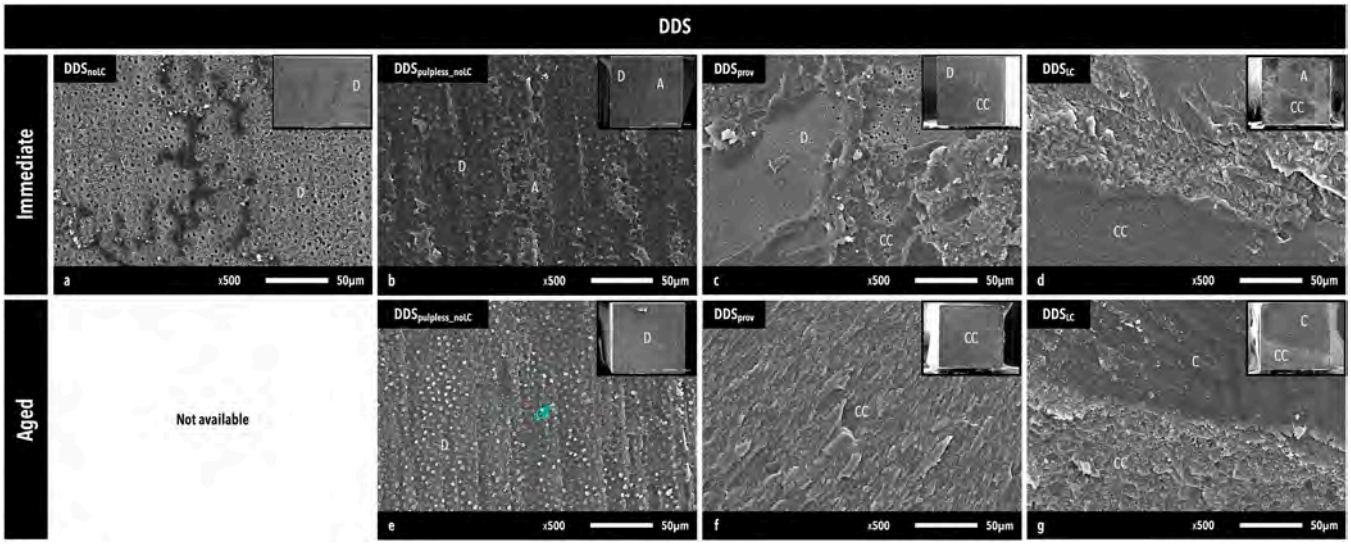

Fig. 3. Representative SEM photomicrographs showing the predominant failure modes in the experimental DDS groups, both 'immediately' and after 'aging'. Typical examples of 'mixed failures' are shown for all groups except for 'DDS_{noLC}' (a) and '$DDS_{pulpless\_noLC}$' (b,e), which display 'adhesive interfacial failures with dentin'. There is no photomicrograph for the 'DDS_{noLC}' aged group because no sample survived the tooth-sectioning process. D: dentin; A: adhesive; CC: composite cement; C: CAD/CAM composite; Hand-pointer: smear-plug.

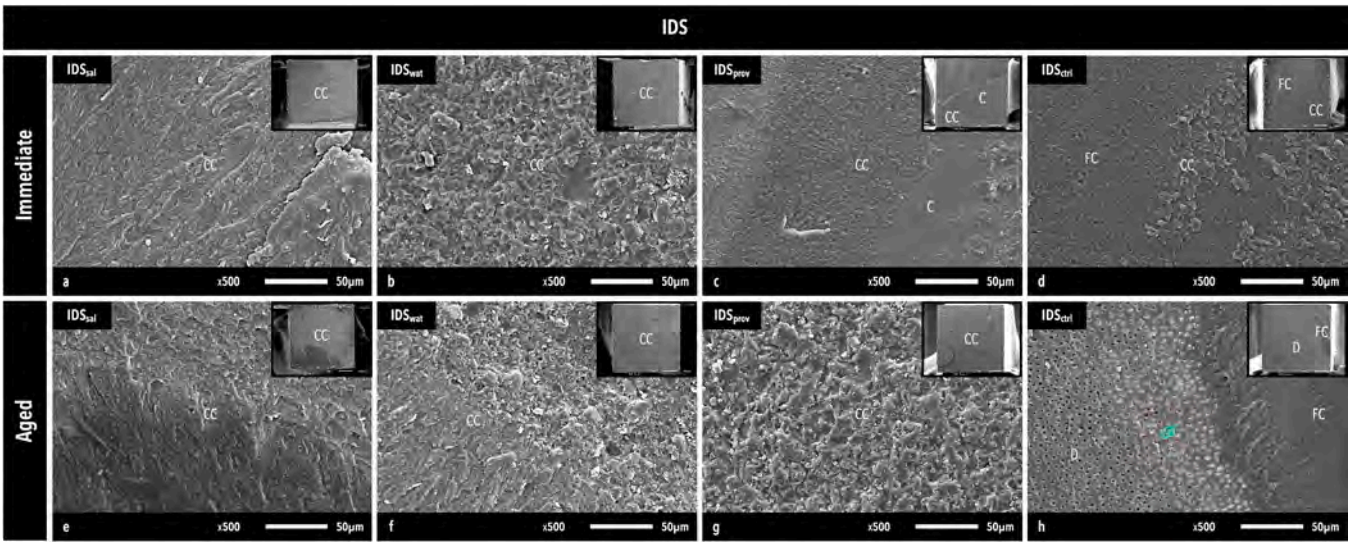

Fig. 4. Representative SEM photomicrographs showing the predominant failure modes in the experimental IDS groups, both 'immediately' and after 'aging'. Predominantly, 'mixed failure' modes were recorded for all groups. D: dentin; FC: flowable composite; CC: composite cement; C: CAD/CAM composite; Hand-pointer: smear-plug.

this study were 5.0 mm thick, the extended light-curing time likely ensured optimal polymerization at the adhesive interface.

Simultaneously curing the adhesive and composite cement can also generate higher polymerization shrinkage stress at the adhesive interface [16,21,45], potentially compromising dentin bond strength [16,38]. In cement spaces beneath semi-direct or indirect restorations, the adhesive and composite cement are confined between restoration and tooth prep. This confinement creates a high C-factor due to the large ratio of bonded to unbonded surfaces [46], amplifying de-bonding stress at the adhesive interface [47]. By separately curing the adhesive before luting, the large free (non-bonded) surface and thus relatively low C-factor conditions allow for shrinkage of the adhesive to occur without imposing excessive stress on the vulnerable bond to dentin.

Endodontic treatment leads to structural and biomechanical changes in teeth, most notably a reduction in water content [48–50]. This dehydration increases the Young's modulus of dentin, making it stiffer but also more brittle and less flexible (stress absorbing) [49]. To better understand the reduced bond strength observed in the 'DDS_{noLC}' group, as mentioned above likely due to water uptake from the underlying dentin at the adhesive interface, simulated pulpless teeth ('$DDS_{pulpless\_noLC}$') were tested in this study. In this group, the adhesive and composite cement were simultaneously light-cured, and the pulp chambers were filled with composite. Although the pulpless teeth were dehydrated and rehydrated, it is important to note that extracted teeth cannot be fully rehydrated and have a drier nature [51]. The '$DDS_{pulpless\_noLC}$' group exhibited significantly lower water content compared to the other experimental groups, which also reduced outward dentinal fluid movement toward the adhesive interface. This differs from the 'DDS_{noLC}' group, where the intact dental pulp and highly permeable dentin facilitated greater water movement. As a result, the '$DDS_{pulpless\_noLC}$' group showed significantly higher bond strength (μTBS) and fewer pre-testing failures (PTFs) compared to the 'DDS_{noLC}' group. This

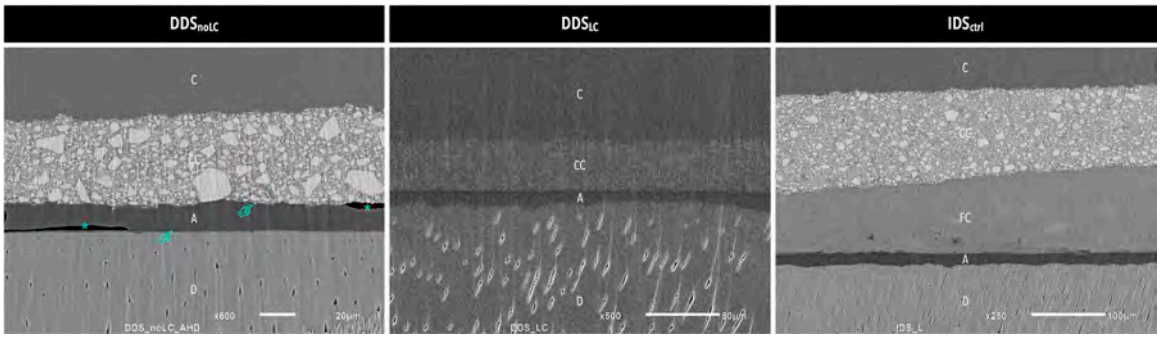

Fig. 5. Photomicrographs showing the argon-ion-beam cross-section polished adhesive interface morphology for three selected experimental protocols: 'DDS$_{noLC}$' (left), showing the DDS protocol without a separately light-cured adhesive-resin layer; 'DDS$_{LC}$' (middle), representing the DDS protocol with an adhesive-resin layer separately light-cured prior to adhesive luting; 'IDS$_{ctrl}$' (right), showing the optimized IDS protocol without contamination. D: dentin; A: adhesive; FC: flowable composite; CC: composite cement; C: CAD/CAM composite; Hand-pointers: cracks; *: voids.

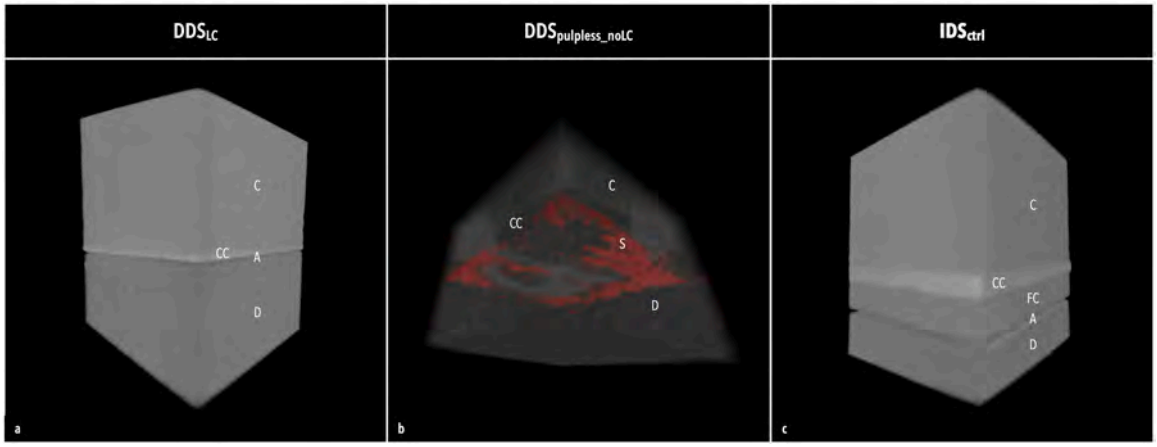

Fig. 6. Micro-CT 3D-reconstruction of micro-specimen segments illustrating the multilayered interfacial structure formed by 'DDS$_{ctrl}$', 'DDS$_{pulpless_noLC}$' and 'IDS$_{ctrl}$'. While no interfacial nano-leakage was observed for 'DDS$_{ctrl}$' and 'IDS$_{ctrl}$', silver particles (S), additionally marked in red, were observed at the 'DDS$_{pulpless_noLC}$' interface, indicating that interfacial gaps enabled silver-particle infiltration. A: adhesive; C: CAD/CAM composite; CC: composite cement; D: dentin; FC: flowable composite; S: silver particles.

finding led to the rejection of the second hypothesis (2) that tooth vitality does not affect bond strength to dentin. It confirms that water uptake from the underlying dentin at the adhesive interface is the most plausible cause of the PTFs observed when the adhesive and composite cement were cured simultaneously.

The slightly reduced μTBS in the 'DDS$_{pulpless_noLC}$' group compared to that recorded in the 'DDS' group, though still higher than that of the 'DDS$_{noLC}$' group (and only three PTFs), may be attributed to the drier and more brittle nature of dentin in pulpless teeth. Unfortunately, this study did not include a group using the 'DDS$_{pulpless_noLC}$' protocol with separate light-curing of the adhesive prior to luting. Given the inclusion of already eight experimental scenarios, adding another group was deemed beyond the scope of this study. It is also worth noting that the pulpless teeth used in the present study do not fully replicate the clinical condition of endodontically treated teeth. Endodontic treatment introduces additional factors, such as irrigation solutions and root-canal filling materials, which may [50,52,53] or may not [53–56] affect dentin bond strength.

A premolar/molar that needs to be restored with a semi-direct or indirect bonded restoration typically has already lost a substantial amount of tooth structure. A large amount of dentin is usually exposed during preparation. The deeper the preparation, the more and wider the dentin tubules are exposed, increasing dentin permeability [57]. Exposed dentin, especially with increased permeability, is vulnerable to bacterial infiltration and external stimuli, raising the likelihood of postoperative hypersensitivity and pulpal irritation [9–11]. To protect the exposed dentin, the immediate application of adhesive, either alone or in combination with a low-viscosity composite layer, was initially introduced as the "dual bonding technique" or "resin coating", later being referred to as IDS [5,9–15,58]. This technique was reported to be effective in protecting dentin, increasing μTBS and marginal adaptation of indirect restorations [12–14,58]. To validate the IDS technique, most *in-vitro* studies however compared the μTBS achieved by an adhesive luting protocol like the 'DDS$_{noLC}$' experimental group in this study with the adhesive (delay-)cured together with the composite, as opposed to IDS with the adhesive light-cured separately and prior to the actual adhesive luting. Generally, these studies find significantly higher μTBS for IDS as compared to the 'DDS$_{noLC}$'-like luting protocol [5,6,45]. Also, some *in-vivo* studies evaluated the benefits of IDS over that of the 'DDS$_{noLC}$'-like luting protocol, confirming again that the better performance achieved with IDS [4,17,18]. A study much earlier conducted by McCabe & Rusby [38] in the 1990's, however, revealed a significantly higher shear bond strength when the adhesive was light-cured separately from the composite, raising concerns about bonding performance if the adhesive is light-cured together with the luting composite. The latter concern was confirmed in the present study, by which the third hypothesis (3a) that IDS would not affect μTBS to dentin, was rejected when comparing 'DDS$_{noLC}$' with all four IDS scenarios. The superior performance of IDS compared to DDS reported in this study ('DDS$_{noLC}$') and in the multiple abovementioned studies should however be attributed to the omission of separately light-curing the adhesive before adhesive luting. In fact, this study revealed that DDS equals IDS in adhesive

luting performance when the adhesive is separately light-cured, by which the third hypothesis (3b) that IDS would not affect μTBS to dentin, was accepted when comparing 'DDS_{LC}' and 'DDS_{prov}' with all four IDS scenarios. Clinically, separately light-curing the adhesive prior to adhesive luting with a composite cement following a chairside one-visit, semi-direct restorative treatment is only possible when the adhesive is sufficiently air-thinned prior to light-curing in order not to impair the fit of the restoration. While universal adhesives commonly have a thin film thickness, often even below 10 μm, even multi-step adhesives that provide a separate adhesive resin can be sufficiently air-thinned without impairing the restoration fit [59], also taking into account a cement space is considered clinically acceptable up to 120 μm [60].

Following a chairside DDS luting protocol, 'DDS_{prov}' exhibited a slightly but significantly higher μTBS compared to 'DDS_{LC}', by which the fourth hypothesis that provisionalization (4a) would not affect μTBS to dentin, should be rejected for DDS. Although this difference may have limited clinical relevance given the modest improvement, the dentin sandblasting performed after provisional removal in the 'DDS_{prov}' protocol likely contributed to the enhanced bonding performance. This improvement can be attributed to additional surface micro-roughening, additional surface cleaning with removal of potential provisional cement remnants, and/or the creation of a thinner, less compact smear layer that interferes less with bonding. A recently published systematic review and meta-analysis revealed that temporary cementation in a DDS scenario without separately light-curing the adhesive (as 'DDS_{noLC}' in this study) significantly reduced bond strength [25]. Non-eugenol temporary cements caused a significant decrease in dentin-bond strength, whereas polycarboxylate and calcium-hydroxide cements did not have a notable impact [25]. Nevertheless, dentin-bond strength was effectively restored when the temporary cement contamination was removed through aluminum-oxide air-abrasion, as was recorded and reported above for the 'DDS_{prov}' scenario in the present study. Regarding the bond-promoting effect of particulate air-abrasion, a recently published scoping review reported no influence on dentin-bond strength in 63.6 % of the studies included in the review, while 30.3 % studies revealed improved bonding performance, versus solely 6.1 % studies that reported decreased dentin-bonding performance [61]. Clear specific protocol recommendations could not be given, considering the large variation in air-abrasion parameters. An improvement was more often noticed for etch-and-rinse adhesives compared to the self-etch adhesives [61,62].

The most common clinical scenario to adhesively lute restorations typically requires two appointments: the first for tooth preparation, digital impressioning, and provisional restoration, and the second for adhesively luting the final restoration [8,25,63]. To replicate this scenario, the groups 'DDS_{prov}' and 'IDS_{prov}' were designed, hereby simulating clinical situations where DDS and IDS surfaces are protected with a temporary restoration after tooth preparation while being exposed to saliva in the patient's mouth for (1-)2 weeks. As worse-case scenario of an indirect luting protocol (negative control), the IDS surface was also left exposed to saliva for two weeks ('IDS_{sal}') without provisional prior to adhesive luting, this considering a clinical case when the tooth prep is solely protected by IDS without provisional (on the condition that no tooth movement is expected during the 10–14 days in between both appointments). A similar but less severe condition involved exposure of the IDS surface to solely water for two weeks ('IDS_{wat}'). The 'IDS_{ctrl}' condition served as the most optimum adhesive luting procedure (positive control). In the present study, the four IDS scenarios resulted in nearly equal adhesive luting performance with solely 'IDS_{wat}' exhibiting a significantly higher μTBS as compared to 'IDS_{ctrl}' and 'IDS_{prov}', for which no plausible explanation can be given. Neither were substantial differences in failure mode recorded. As no significant difference in μTBS was recorded for 'IDS_{prov}' compared to 'IDS_{ctrl}', the fourth hypothesis that provisionalization (4b) would not affect μTBS to dentin, was accepted for IDS.

When IDS is followed by cementation of a provisional, the IDS layer is contaminated with provisional cement. Several studies have assessed the impact of IDS-surface contamination with different temporary cements and materials on dentin-bond strength [25,44,58,63–67], while other studies have evaluated the impact of different IDS surface-cleaning methods on bond strength [68,69]. Resin-based temporary materials and cements should be avoided after IDS, because they may reduce bond strength unless the IDS surface is covered with a separation medium, such as water-soluble gel or petroleum jelly [5,65]. In general, luting a temporary restoration on an IDS surface does not appear to affect bond strength when a proper cement-removal method is employed [25,44,59,63,65,67,68], as also appeared in this study. Alumina air-abrasion followed by 37 % phosphoric-acid application was considered the preferred IDS-surface treatment in several studies [68,69]. The concept of cleaning the IDS surface is, at least in part, based on surface-treatment techniques used for resin-composite restoration repair [70,71]. A combination of mechanical (roughening the surface by particulate air abrasion) and chemical (silane, adhesive application) pretreatments resulted in superior repair bond strength of resin-based composites tested under both short- and long-term simulated aging conditions [71]. In the present study, silane was not applied on the IDS surface. According to a systematic review of Mendes *et al.* [72], additional silanization nevertheless increased the repair bond strength of methacrylate resin-based composites. Further research is recommended to evaluate the impact of separate silane application on the IDS surface as crucial step in the adhesive luting process of an indirect restoration.

To stress the adhesive interface, the teeth restored using various adhesive luting protocols were subjected to mechanical fatigue with 1,200,000 chewing cycles, followed by exposure of the μ-specimens to 10,000 thermocycles, as was also implemented in other similar studies [32,33]. Previous studies estimated that 1,200,000 chewing cycles would correspond to approximately five years of clinical service [73,74], while 10,000 thermocycles would equate to one year of clinical use [75]. Despite the relatively rigorous aging protocol, the general study outcome remained largely unchanged, as no significant difference in μTBS was observed in 5 out of the 8 experimental scenarios upon aging. Based on these results, the fifth hypothesis (5) that aging would not affect μTBS to dentin, was accepted for the three DDS and two IDS conditions. However, a slightly but significantly lower μTBS was observed upon aging for '$DDS_{pulpless\_noLC}$' and 'DDS_{prov}', while a slightly but significantly higher μTBS was recorded for 'IDS'. Although the latter findings required rejecting the fifth hypothesis for these conditions, the observed differences are unlikely to have major clinical relevance due to their minimal magnitude. Also, the failure-mode distribution did not change much for most experimental IDS and DDS scenarios, with the exception of '$DDS_{pulpless\_noLC}$', for which upon aging more adhesive interfacial failures with dentin were observed.

In general, the bonding interface tends to degrade over time, primarily due to water sorption at the adhesive interface [76–78]. This process contributes to resin hydrolysis and collagen breakdown, both of which adversely affect bond strength [76–78]. Since adhesive hydrophilicity is closely linked to hydrolytic degradation [77,78], using different adhesives while following the same bonding protocol as in this study could potentially produce different results.

SEM of the adhesive interface morphology revealed interfacial porosities and cracks at both the adhesive-dentin and adhesive-cement interfaces for 'DDS_{noLC}', hereby confirming previous research mentioned above. These porosities were likely caused by water sorption from the underlying dentin when the adhesive resin was delay-cured together with the composite cement, explaining also the 100 % PTFs and the 100 % 'adhesive interfacial failure with dentin' mode observed in this study. In contrast, tight multi-layer interfaces without interfacial porosities were observed when the adhesive was separately light-cured, either in an optimized DDS ('DDS_{LC}') or IDS ('IDS_{ctrl}') scenario.

Strong air-thinning of the C-SE2 bonding layer, combined with the use of a restorative composite as luting agent, resulted in a prep-restoration cement space of approximately 45 μm for the 'DDS_{LC}'

protocol. This included an adhesive layer of about 10 μm thick and a (luting) composite-resin layer of around 35 μm. This cement space aligns well with the clinically accepted range of up to 120 μm [60,79], still enabling a thicker cement space. The cementation procedure in this study involved applying a consistent weight of 1 kg for 1 min to standardize the cement thickness. However, this method does not perfectly simulate clinical conditions, where restorations are typically placed using finger pressure until the initial light-curing step. By contrast, the IDS protocol yielded a cementation space of approximately 230 μm, comprising an adhesive-resin layer of about 15 μm, an IDS flowable composite layer of roughly 100 μm, and a (luting) composite-resin layer of around 120 μm.

Micro-CT interfacial analysis did not reveal interfacial nano-leakage for 'DDS_{LC}' and 'IDS_{ctrl}', confirming that a stable and reliable interfacial integration with dentin was achieved following these two adhesive luting scenarios, in full agreement with all other data recorded in this study for both scenarios. The interfacial nano-leakage observed for '$DDS_{pulpless\_noLC}$' points to a weaker interfacial interaction with dentin, as appeared also from the significantly lower μTBS measured for this adhesive luting scenario. Notably, the '$DDS_{pulpless\_noLC}$' scenario most closely resembles the 'DDS_{noLC}' adhesive luting scenario, of which the μ-specimens could even not be processed for μCT examination.

Except for 'DDS_{noLC}', and '$DDS_{pulpless\_noLC}$' to a less extent, the other two DDS and four IDS protocols to adhesively lute composite CAD/CAM restorations in the present study enabled strong and reliable bonding to dentin. Note that the C-SE2 (Kuraray Noritake) was applied in a slightly different manner than recommended by the manufacturer. Instead of leaving it undisturbed for 20 sec as instructed, the C-SE2 primer was brushed in an active motion for the same duration. Some *in-vitro* studies showed that continuous agitation of the primer during application results in a better resin infiltration and higher dentin-bond strength [57,80].

In conclusion, contrary to the outcomes repeatedly reported in multiple systematic reviews evaluating the effect of IDS versus DDS on adhesive luting performance, as measured in terms of dentin-bond strength, IDS does not outperform DDS when the adhesive is separately light-cured before adhesively luting the restoration, as per the chairside 'DDS_{LC}' protocol investigated in this study. Obviously, an IDS protocol remains the preferred choice when preparation undercuts need to be blocked out or when the tooth is restored indirectly over two appointments. However, this study also demonstrated that 'DDS_{LC}', thus by separately light-curing the adhesive to stabilize the adhesive interface prior to actual luting, can also be effectively used when a provisional restoration can be securely retained with temporary cement, offering dentin protection and preventing postoperative sensitivity. In cases of non-retentive preparations, retention of the provisional can be challenging [81], where an IDS procedure is again recommended. Ultimately, the choice between DDS ('DDS_{LC}') and IDS lies with the operator, who should select the approach that best suits the clinical circumstances as well as the practitioner's expertise.

A key limitation of this laboratory study is that it does not fully replicate real clinical conditions. The use of third molars from young patients, exhibiting highly permeable dentin, represents a worse-case scenario that may exaggerate the effects of water sorption at the adhesive interface. In actual clinical practice, restorations are often bonded to less permeable, more sclerotic or previously restored dentin, which may result in different bonding. Additionally, despite efforts to simulate clinical procedures, factors such as finger pressure during restoration seating, patient-related variables like pulpal pressure and moisture control, and the long-term effects of intraoral aging cannot be completely reproduced in the lab. Therefore, while the study provides valuable insights into the bonding protocols studied, its results should be considered as directional and in principle confirmed through randomized controlled clinical trials, which provide a higher level of evidence.

Declaration of Competing Interest

None.

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