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A Polymer-Infiltrated Ceramic as Base Adherent in an Experimental Specimen Model to Test the Shear Bond Strength of CAD-CAM Monolithic Ceramics Used in Resin-Bonded Dental Bridges

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Abstract: Traditional load-to-failure tests fail to recreate clinical failures of all-ceramic restorations. Experimental fabrication, similar to prosthetic laboratory and clinical procedures, best predicts future clinical performance. A hybrid ceramic adherend, mechanically similar to a human tooth, was tested by comparing the shear bond strength (SBS) and fracture mode of four restorative materials adhered with a dual-cure adhesive cement. Surface energy, shear bond strength (SBS), and fracture mode were assessed. Vita Enamic (ENA), Vita Suprinity (SUP), Vita Y-TPZ (Y-ZT), and a nanohybrid composite (RES) (control group) cylinders, adhered with RelyX Ultimate to ENA blocks were assembled in experimental specimens simulating a 3-unit resin-bonded dental bridge. The ENA adherend was ground or treated with 5% hydrofluoric acid for 60 s. Monobond Plus was used as the coupling agent. Mean shear stress (MPa) was calculated for each group. Forest plots by material elaborated after calculating the difference in means and effect size ($\alpha = 0.05$; 95% CI; Z-value = 1.96) revealed significant differences in the shear force behavior between materials ($p < 0.01$). RES (69.10 ± 24.58 MPa) > ENA (18.38 ± 8.51 MPa) > SUP (11.44 ± 4.04 MPa) > Y-ZT (18.48 ± 12.12 MPa). Y-ZT and SUP exhibited pre-test failures. SBS was not related to surface energy. The failure mode in the Y-ZT group was material-dependent and exclusively adhesive. ENA is a potential adherend for dental materials SBS tests. In this experimental design, it withstood 103 MPa of adhesive stress before cohesive failure.

Keywords: adhesive stress; bonding; hybrid ceramic; CAD-CAM; resin cement; resin-bonded bridge; shear bond strength; surface energy; surface treatment; zirconia-reinforced lithium disilicate; yttria-stabilized tetragonal zirconia

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1. Introduction

Computer-aided design-computer-aided manufacturing (CAD-CAM) materials are versatile and emerging as the materials of choice for many restorations. However, proper

clinical and research-based evidence is required to confirm their success and durability before they can be recommended for patient care [1–3]. Scientific evidence is mainly based on laboratory tests. It has long been suggested [4] that traditional fracture tests of single-unit all-ceramic prostheses are inappropriate because they do not mimic the failure mechanisms observed in retrieved failed clinical specimens. Current evidence suggests as best predictors of future clinical performance are tests with full anatomy restoration design, interproximal wall-length variations, core shape and veneer thickness similar to clinical design, fabrication procedures following laboratory and clinical procedures, comparison of support structures present in the clinical context (e.g., implant- vs. dentin-supported), and fatigue load assessment in water with sliding contacts [5]. In addition, in vitro studies frequently do not simulate bruxism scenarios that often occur in vivo [6].

The high innovation rate of CAD-CAM materials and technology requires good knowledge for optimal and successful clinical use [7]. In a clinical, laboratory, or centralized environment, workflow options are endless, and the variety of technologies is vast, with increased levels of communication, predictability, productivity, efficiency, and patient care [8]. While subtractive techniques are primarily used for definitive restorative purposes, additive techniques, mainly used for treatment planning or temporary devices, offer potential material savings and are beneficial when creating complex geometries, with the disadvantages of high cost, time-consuming post-processing, low flexural strength, and lack of long-term clinical research essential to facilitate the translation of its applications from laboratory to clinical setting [9–11]. CAD-CAM monolithic ceramics aim to avoid the technical and mechanical issues associated with layered fixed prostheses and apparently have high survival and low complication rates [12,13]. Further randomized controlled trials (RCTs) are needed to evaluate their long-term clinical performance compared to veneered restorations [12]. Experimental in vitro research designs that simulate clinical conditions using a polymer-infiltrated ceramic as a standardized adherent to replace natural teeth can help to understand the behavior of materials and prostheses. To date, few experimental protocols have been transposed directly from laboratory studies to clinical contexts [1]. Laboratory tests performed on natural teeth have inherent biological variability, with implied heterogeneity of results, and confront ethical restrictions. Testing adhesive protocols brought from the clinic to the laboratory and not vice versa can contribute to clarifying the effectiveness of adhesive procedures in the clinical context as a part of rehabilitative treatment. Adhesive restorations rely on bonding systems to form micromechanical bonds with the teeth [14,15]. However, chemical interactions may occur between functional monomers and components, with potential benefits [16–18].

Resin-based cement is widely used to adhere to nonmetallic restorations. Bond strength tests are essential to study mechanical performance [16], as mechanical, thermal, and passive hydrolysis may occur in the mouth, resulting in loss of adhesive joint performance [19]. CAD-CAM esthetic materials fall into four main classes: glass-matrix ceramics, polycrystalline ceramics, indirect composites, and hybrid ceramics [20,21].

The CAD-CAM hybrid ceramic Vita Enamic (ENA) (VITA Zahnfabrik, Bad Säckingen, Germany) is based on a dominant ceramic network reinforced with an acrylic polymer network resin [20]. It combines a low flexural modulus with high flexural strength (150–160 MPa), which is expected to increase its ability to withstand loads by undergoing more elastic deformation before failure, similar to the behavior of human teeth [22]. The typical double-network microstructure of the ENA is essential for the micromechanical bonding and performance of the adhesive interface [23] owing to the decrease in crack propagation [24].

Vita Suprinity (SUP) (VITA Zahnfabrik, Bad Säckingen, Germany) is a versatile precrystallized zirconia-reinforced lithium silicate ceramic with easy milling and polishing. It has a fine-grained (0.5–0.7 μm) and homogeneous structure, with a consistently high load capacity (flexural strength, crystallized at 420 MPa) [25]. Despite the biocompatibility and mechanical properties of SUP, data are still scarce, often controversial, and limited to short-term observational periods, which require further in vitro/in vivo studies primarily for

long-term performance [26]. The polycrystalline ceramic Vita YZ HT (Y-ZT) (VITA Zahnfabrik, Bad Säckingen, Germany) is a tough opaque whitish material [27,28], and its physical and mechanical characteristics have been used as references for new generations (high flexural strength of 1200–1500 MPa) [27,28]. Recent compositions with higher yttria content, while improving zirconia esthetically, sacrifice mechanical performance, making it more susceptible to breakage [29–31]. These findings suggest caution when extrapolating results from longevity research focusing on older materials [7] despite promising RCTs results [32]. The bonding ability of zirconia is conditioned by airborne particle abrasion and primers or adhesives containing 10-methacryloxydecyl dihydrogen phosphate (MDP) [1,33].

Retention of restorative materials depends on the quality of the adhesive joint, which determines the bond quality at different interfaces [3]. The interface between the cement and dental tissue is essential, and the connection between the cement and the surface of the restorative material also plays a crucial role [3,34,35]. This process involves adhesion and cohesion [35–37], the first between substrates and the second within each substrate. CAD-CAM restorative materials require a multistep bonding procedure, and the specific bonding strategy for each material is determined based on its composition [1,3,20,38,39].

Characterizing the adhesive interface before adhesion, during function, and after failure is helpful for investigations and remains a significant challenge [36]. The surface treatment of each CAD-CAM material and the luting resin affect the adhesion bond strength. Therefore, a specific adhesive cementation protocol is required for each pair of materials to achieve the highest bond strength [1,40,41]. The adhesive strength or efficacy is influenced by the amount of light transmitted through the resin-matrix composite cement, which in turn is influenced by the size, content, microstructure, and shape of the inorganic filler particles.

A decrease in the degree of conversion negatively affects the physical and mechanical properties of resin-matrix composites [42]. Optimal light-curing parameters result in a low release of monomers and minimal toxicity to the dentin-pulp complex, mucosa, or periodontal tissues [34,43,44]. This aspect is pertinent because the release of these monomers must be added to that released from the restoration itself whenever a resin-based CAD-CAM material is used, except for Vita Enamic (ENA) [45], probably because of its particular structure. For these reasons, a dual-cured adhesive cement, RelyX Ultimate (RU) (3M ESPE, Seefeld, Germany) with a 3-steps adhesive strategy under photoactivated polymerization was used to assemble the experimental model [36,46,47]. Advances in adhesive dentistry and technology have expanded the possibility of using resin-bonded bridges (RBB) with alternative preparation designs and materials [48,49].

This study aimed to test a hybrid ceramic as an adherend for shear bond tests in an experimental specimen model. In parallel, the model was used to evaluate the mechanical behavior of four materials, of which three CAD-CAM monolithic ceramics, potential materials to rehabilitate clinical maxillary lateral incisor agenesis situations. The null hypotheses were that the hybrid ceramic was not a mechanically suitable adherend for shear bond tests and that no differences would be found in the mechanical behavior between the CAD-CAM monolithic ceramics.

2. Research Significance

To the best of our knowledge, this experimental model is innovative because it uses an industrially produced material as an adherend from which a uniform composition is expected, unlike what happens with biological materials. Its hybrid constitution gives it a mechanical behavior that is hypothetically similar to that of a human tooth [22]. This makes it a candidate adherend for future adhesive strength tests of dental materials, at least in preliminary studies. This experimental model identified significant differences between the restorative materials. This would overcome the ethical constraints and result biases inherent in the use of biological materials.

3. Materials and Methods

The primary materials used in this study are listed in Table 1.

Table 1. General description of the materials used in this study, their compositions, and manufacturers.

Material	Name	Code	Composition	Manufacturer
CAD-CAM Ceramics	VITA Enamic	ENA	86% feldspar ceramic: SiO_2 58%–63%, Al_2O_3 20%–23%, Na_2O_9–11%, K_2O_4–6% by weight, 14% polymer by weight: TEGDMA, UDMA	VITA Zahnfabrik, Bad Säckingen, Germany
	VITA Suprinity	SUP	Zirconium oxide 8–12, silicon dioxide 56%–64%, lithium oxide 15%–21%, various > 10% by weight	VITA Zahnfabrik, Bad Säckingen, Germany
	VITA 3Y-TPZ	Y-ZT	Zirconia reinforced with 3% Yitria	VITA Zahnfabrik, Bad Säckingen, Germany
Resin-matrix restorative composite	PROCLINIC EXPERT Nano Hybrid composite	RES	22.5% weight, multifunctional methacrylic ester; 77.5% weight, inorganic filler (40 nm–1.5 microns).	SDI Limited, Burnston, AUS
Resin-matrix composite cement	RelyX Ultimate	RU	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond™ copolymer filler, ethanol, water, initiators, silane	3M Oral Care, St. Paul, MN, USA
Etching agent	VITA ADIVA Cera Etch	HF5	Hydrofluoric acid 5%	VITA Zahnfabrik, Bad Säck ingen, Germany
Ceramic primer	Monobond Plus	MB	50%–100% ethanol, disulfit methacrylate, ≤2.5% phosphoric acid di methacrylate, ≤2.5% 3-trimethoxysilylpropyl methacrylate	Ivoclar Vivadent AG, Schaan, Liechtenstein
	VITA ADIVA C Primer	CP	Solution of methacrylsilanes in ethanol	VITA Zahnfabrik, Bad Säck ingen, Germany
Adhesive system	Scotchbond Universal adhesive	SB-U	MDP, Bis-GMA, phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane-treated silica	3M Oral Care, St. Paul, MN, USA

VITA Enamic hybrid ceramic (ENA) blocks were used as base adherents for mechanical tests. The idealized testing protocol is shown in Figure 1. Adhesive protocols and equipment accessible in a clinical context were used instead of sophisticated equipment or chemically aggressive but efficient adhesive protocols frequently found in the literature [1]. Nevertheless, standardization was guaranteed, and a single clinical expert performed all the procedures.

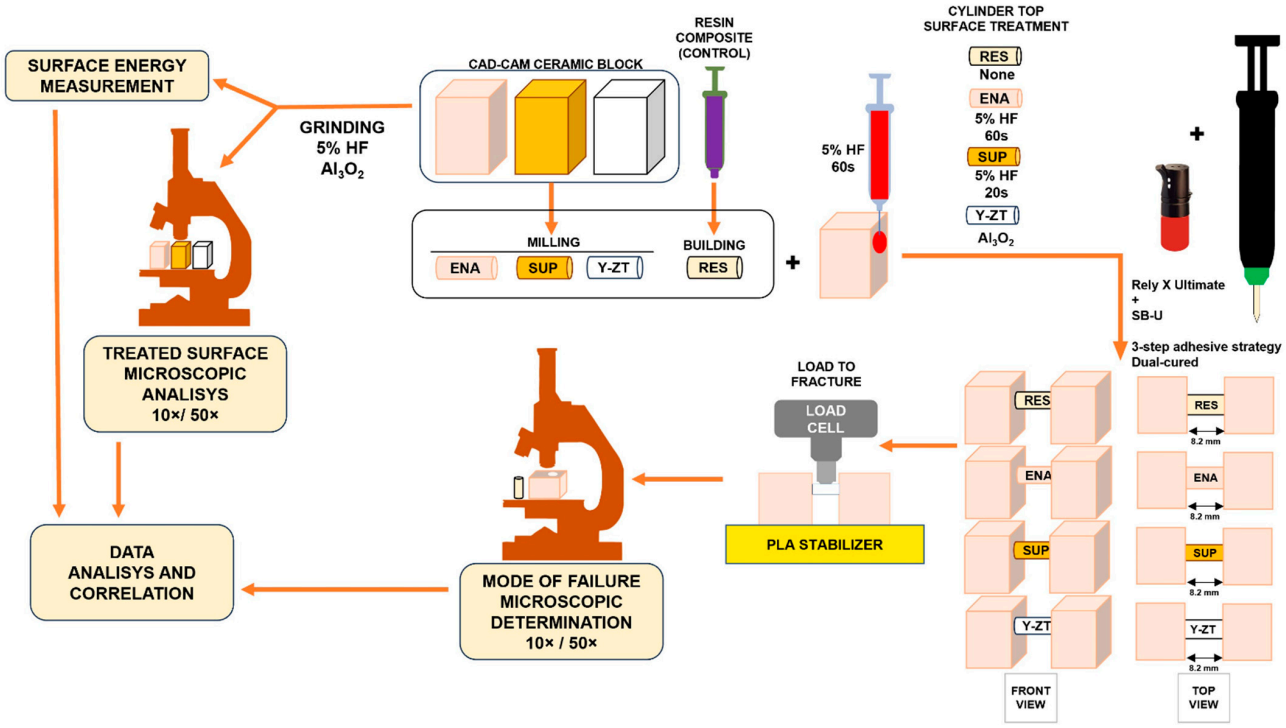


Figure 1. Comprehensive scheme of the experimental protocol workflow. ENA, Vita Enamic; HF, hydrofluoric acid; PLA, polylactic acid; SB-U, Scotchbond Universal adhesive; SUP, Vita Suprinity; Y-ZT, Vita zirconia.

3.1. Preparation of the Bases Adherend

After removing the metallic support pin from the ENA ceramic block (Figure 2A,B), the superficial gloss was removed by dry grinding to simulate the removal of the aprismatic enamel or external fluorohydroxyapatite layer. A coarse finishing disk (Soflex Disc Pop-On, 3M, Saint Paul, MN, USA) mounted in a low-speed handpiece set at 20,000 rpm and attached to the dentist chair at an angle of ±45° with the surface of the block was used. The applied force was driven by hand, as in a clinical setting, by the same restorative dentist (single operator) with >30 years of clinical experience. A new disk was used for each block with eight grinding repetitions. A 20-s oil-free air/water spray removed the debris. The prepared blocks were shuffled to ensure randomization and operator blinding.

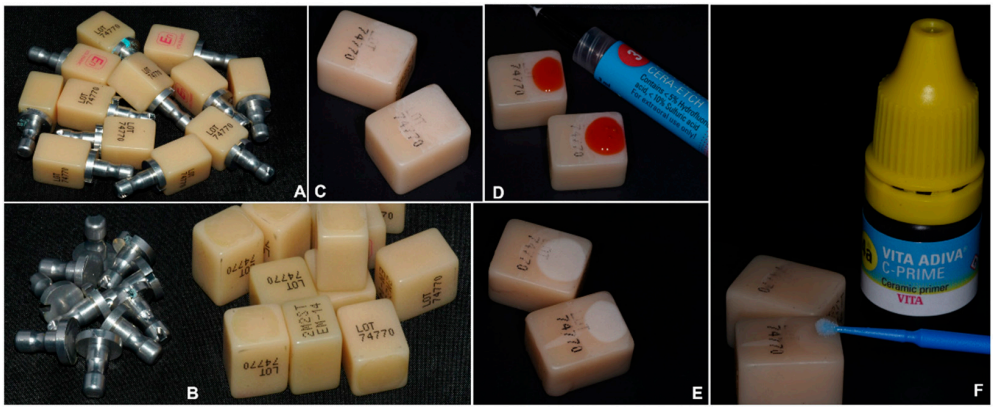


Figure 2. Vita Enamic blocks and surface treatment in the control group (RES). (**A**) before and (**B**) after support pin removal; (**C**) immediately after grinding; (**D**) during the hydrofluoric acid conditioning; (**E**) whitish conditioned and dried surface; (**F**) during the ceramic primer application, as recommended by the manufacturer.

The bonding surface of the ENA block was prepared for bonding following the sequence shown in Figure 2C–E and Figure 3. For standardization, only 4.9% (5%) hydrofluoric acid etching gel from the VITA ADIVA kit (HF5) (Vita Zahnfabrik, Bad Säckingen, Germany) was used to etch all acid-sensitive surfaces involved in the study (20 s, SU group; 60 s, ENA group), according to the manufacturer's instructions. The blocks were etched in pairs to prevent over-etching, and the etching time was controlled using a stopwatch. The treated surfaces were thoroughly cleaned using oil-free water spray for 20 s and then dried using oil-free compressed air for 10 s. The hybrid ceramic was primed with Vita ADIVA C-Primer (Vita Zahnfabrik, Bad Säckingen, Germany) in the control group (Figure 2F), and with a universal silane-containing primer (Monobond Plus, Ivoclar Vivadent) in the other groups [39,50] (Figure 3). Primers were applied using a microbrush and allowed to react for 60 s. If not completely dried after 60 s, air-drying was performed using an oil-free spray.

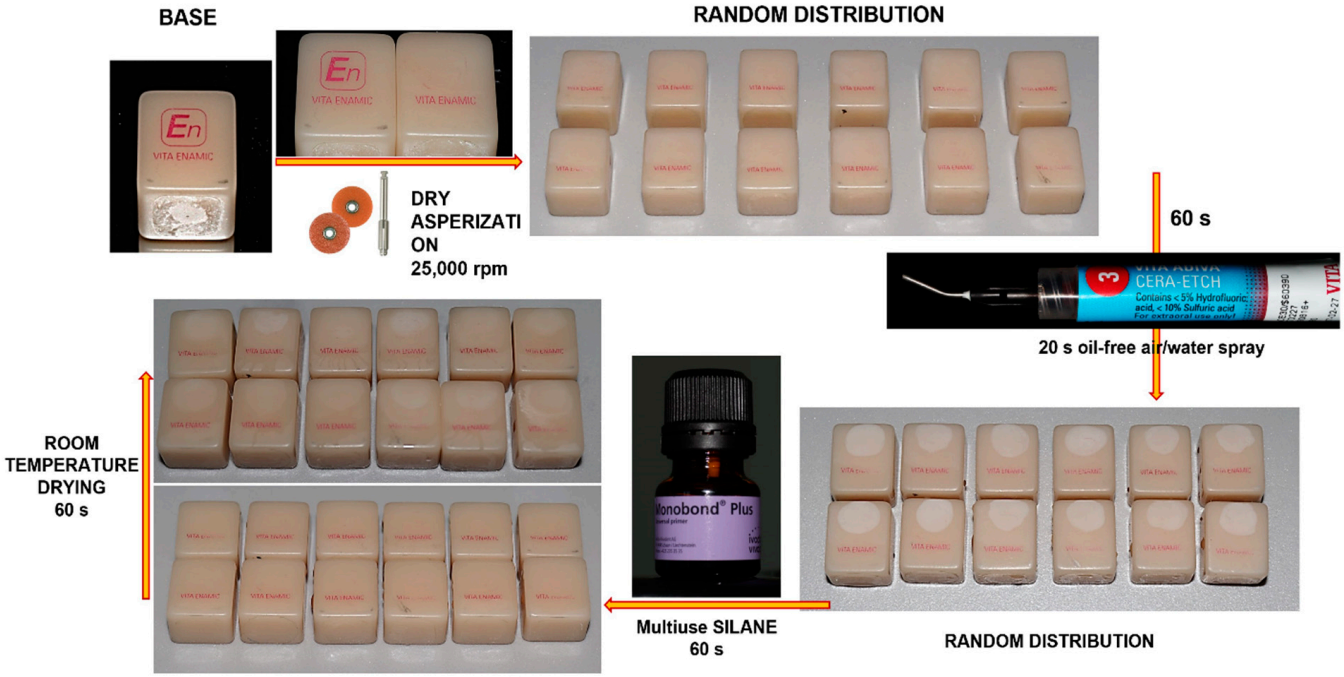


Figure 3. Sequence of the protocol for preparing the specimen bases for the groups ENA, SUP, and Y-ZT.

3.2. Preparation of the Cylinders

Monolithic ceramic cylinders (base diameter, 3.88 mm; length, 8.2 mm) (Abase = $\pi \times r^2$ = 12.19 mm^2), designed with EXOCAD software (Exocad GmbH, Darmstadt, Germany) and fabricated using a CAD-CAM inLab milling machine (Dentsply/Sirona, Charlotte, NC, USA), were produced for each ceramic material (n = 18) (Figure 4). Cylinders of the same dimensions, made of a PROCLINIC EXPERT resin-matrix nanohybrid composite (RES) (SDI Limited, Burnston, Australia) (control group), were manually manufactured using a polycarbonate cylinder template. All cylinders were checked at 10× magnification for cracks, surface discontinuities, and air bubbles and voids in the specific case of the manufactured cylinders.

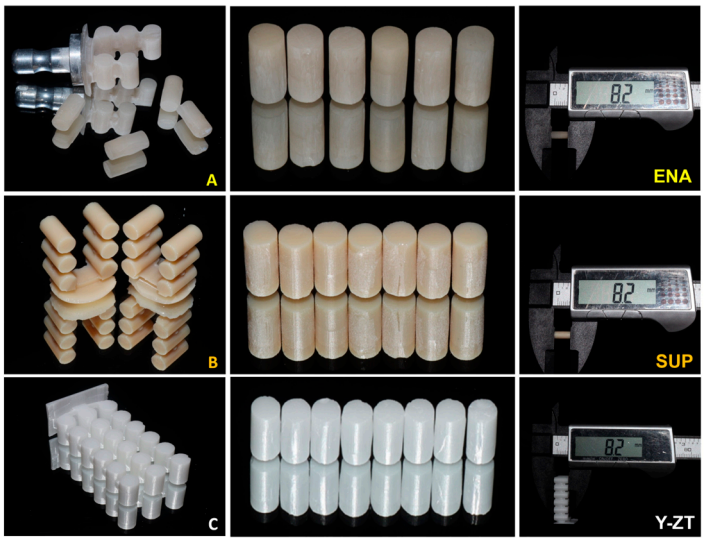


Figure 4. Fabrication and calibration of the CAD-CAM monolithic ceramic cylinders. (**A**) immediately after milling, (**B**) after being cut and regularized, and (**C**) after testing length calibration.

Cylinders considered appropriate for testing were selected (RES, SUP, Y-ZT, n = 5; ENA, n = 6) for bonding with RelyX Ultimate (RU), according to the manufacturer's instructions (Figure 5). The bonding procedures were performed immediately after each surface-conditioning method to avoid surface contamination.

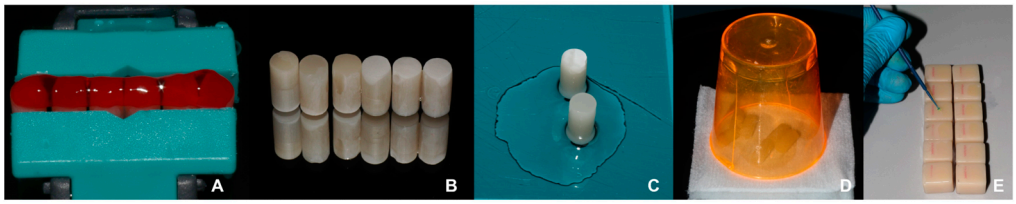


Figure 5. Cylinders and blocks in preparation for adhesion. (**A**) cylinders being conditioned; (**B**) unconditioned (3 on the left) and HF5 conditioned cylinders (3 on the right); (**C**) cylinders immersed in Monobond Plus for 60 s; (**D**) cylinders protected from daylight after adhesive system application; (**E**) adhesive system application on the blocks.

3.3. Specimens Assembling for Shear Strength Test

To allow standardization during specimen assembly, a silicon mold was prepared to accommodate the blocks and allow the exact height of the cemented cylinder between the blocks (Figure 6A–E). Two blocks sustained in a plastic holder by a metallic pin were inserted into a silicone ice cube mold filled with silicone putty. An extra 0.3 mm space was calculated relative to the cylinder length for easy cylinder insertion after cement application on the tops (Figure 6E).

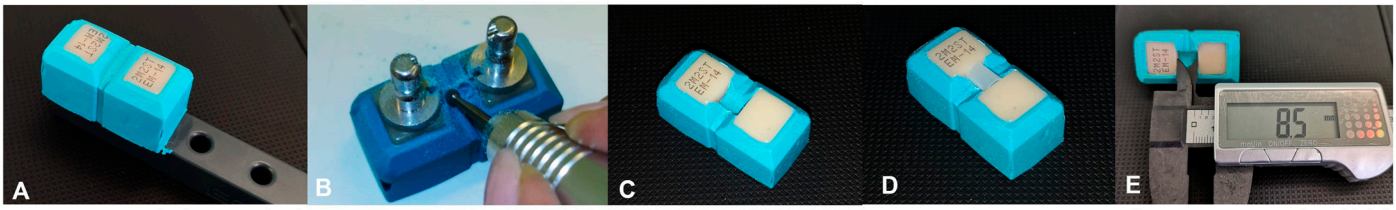


Figure 6. Steps for silicone mold production. (**A**) silicone after setting; (**B**) making the groove to fit the cylinder. Performed with a bladed round drill mounted on a handpiece at low speed; (**C**) detail of the groove definition; (**D**) polycarbonate cylinder template accommodated in the groove for calibration; (**E**) confirming the intended length between the blocks.

The excess cement was immediately removed using a microbrush. A constant pressure (0.5 kg) was applied during cement polymerization using a pinch spring (Figure 7A–C). The interfaces were light-cured for 10 s through each block and then in the middle of the cylinder for a total of 30 s using an Elipar S10 curing unit (1200 mW/cm^2; 3M ESPE) within the accepted procedure [30]. Radiant exposure was ensured by prior calibration of the light-curing device using a radiometer. The compressive force was maintained for 10 min, leaving the material to self-cure.

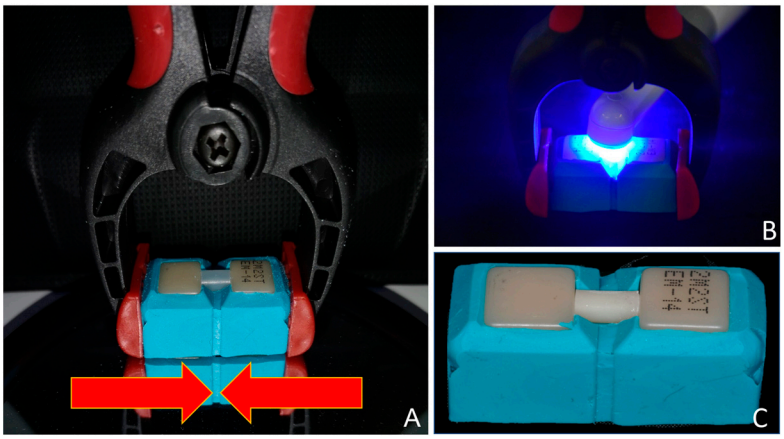


Figure 7. Constant compression for component adaptation. (**A**) during the 10 min; (**B**) detail of the photopolymerization step through the cylinder; (**C**) after polymerization and before mold removal.

Any residual cement was removed using a fine-point sickle scaler (SM 11; Hu-Friedy Co., Chicago, IL, USA). The bonded specimens were stored in saline water for 48 h at 37 °C before the SBS test.

3.4. Mechanical Characterization of Adhesive Joints

To avoid bending during testing, CAD-CAM technology creates a polylactic acid (PLA) base using free-design software and a home-mounted 3D printer. This material was selected because of its properties (environment-friendly option, low melting point (can be printed at lower temperatures and with less energy), ease of use, minimal post-processing, good surface finish, and stiff material) [51]. The details are presented in Figure 8.

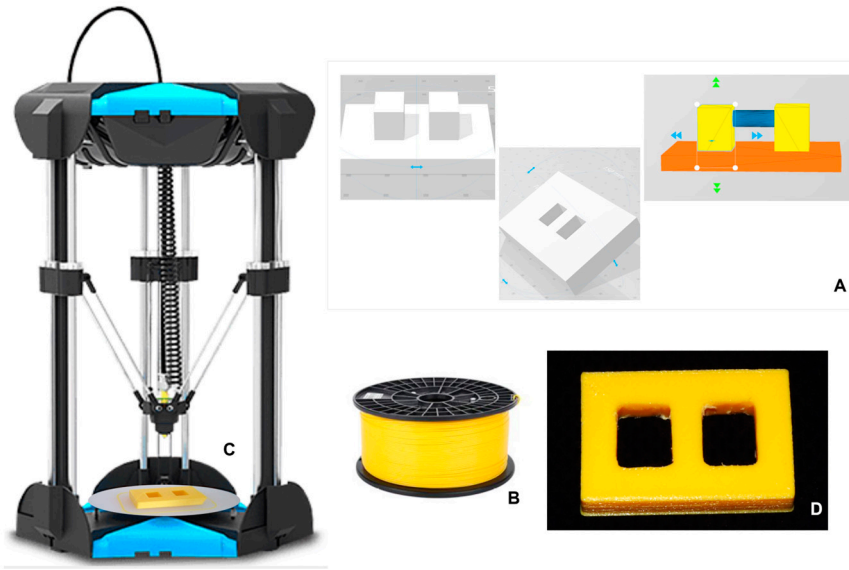


Figure 8. Steps of the polylactic acid (PLA) base design and fabrication. (**A**) design steps; (**B**) PLA coil; (**C**) PLA base in the printing process; (**D**) PLA base just printed.

3.5. In Vitro RBB Materials Adhesive Joint Mechanical Characterization

The specimens of the three CAD-CAM monolithic ceramics, potential candidates for RBB to rehabilitate one missing anterior tooth, were mechanically assessed under load displacement of 0.2 mm/min (Instron–Universal tensile machine) (Figure 9). Load-displacement curves were recorded during the mechanical test. The maximum load in the test was used to identify the experimental RBB setting that supported the highest shear stress and the highest shear stress supported by the adherend before cohesive failure.

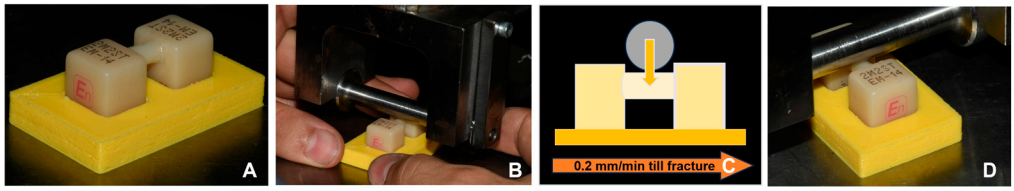


Figure 9. Specimen mounted on the polylactic acid (PLA) base ready for mechanical testing. (**A**) specimen inserted in the PLA base; (**B**) specimen being positioned on the platform; (**C**) basic scheme of the test; and (**D**) initial contact with the specimen and data registration.

3.6. Surface Energy Measurements

The surface energy of each CAD-CAM ceramic by treatment surface was measured to be correlated with the shear strength. The most commonly used surface treatments in the literature were chosen to characterize the surface energy of three CAD-CAM ceramics: grinding, hydrofluoric acid (5%), and aluminum oxide sandblasting [1,34,52].

The measurement protocol followed industry-standard methodology [37]. The surface energy (SE) for each surface treatment was calculated based on the mean of three evaluations for each liquid, using a contact angle goniometer (OCA 15, DataPhysics Instruments GmbH, Filderstadt, Germany). Contact angle measurements were performed under ambient conditions using three different liquids: water (polar liquid), ethylene glycol 55% (polar liquid), and n-hexadecane (nonpolar), following the OWRK method [35]. Figure 10 shows the SE determination after block grinding.

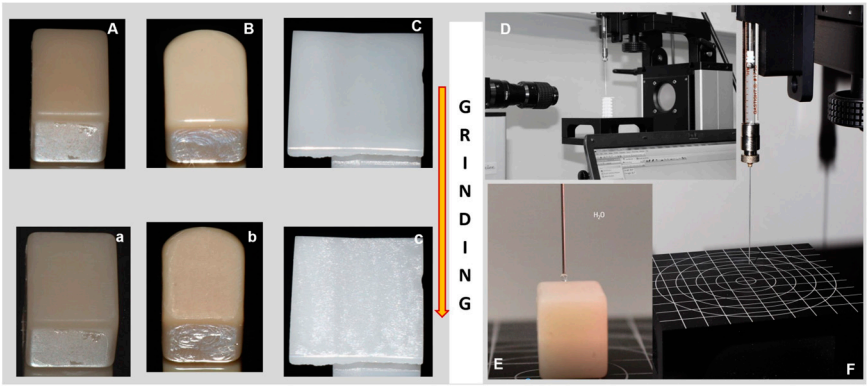


Figure 10. Substrates and equipment used for surface energy determination. (**A**,**a**) Enamic; (**B**,**b**) Suprinity; (**C**,**c**) Y-ZT blocks, as provided and after grinding; (**D**) Y-ZT block positioned for measurements; (**E**) 1 µL of water dropping on a ground Enamic block; and (**F**) detail of the reference platform and injection system.

For only grinding and hydrofluoric acid, the protocols for surface treatment were the same as in subSection 3.1. (Preparation of the base adherend). For the sandblasting surface treatment, the blocks were air-abraded at 0.20 MPa, for 10 s, with 50 µm alumina (Al_2O_3) particles. The nozzle was kept perpendicular and as perpendicular as possible to the surfaces of the blocks (angle between 80° and 90°) at a distance of 10 mm. Air abrasion was

performed with erratic circular motions to ensure an even application of the AIRSONIC Alu-Oxyd powder (Hager-Werken and AZDENT sandblaster, Duisburg, Germany).

3.7. Adhesive Joint Fractography

After SBS testing, the failed specimens (block and cylinder) were inspected at a magnification of 50× and 100× using a digital microscope (AmScope Industrial Inspection, Microscope, United Scope LLC, Irvine, CA, USA). For the mode fracture classification, all components were registered as follows: adhesive fracture (bond failure at the interface between the adhesive and restorative material, even if present in small amounts), cohesive fracture in the block (fracture occurring entirely within the block structure), cohesive fracture in the cylinder (fracture occurring entirely within the restorative material), and mixed when the adhesive and cohesive modes coexisted in the central area of any interface.

3.8. Data Analysis

All data were analyzed to verify the achievement of the proposed goals. The mean load to fracture (N) and shear stress (MPa) with standard deviation (SD) were calculated for each group. A meta-analysis was conducted using the type of CAD-CAM restorative material to evaluate the shear stress between materials after calculating the difference between means and effect sizes (random-effects model; $\alpha = 0.05$; 95% CI; Z-value = 1.96). The failure mode was determined by microscopic observation and was correlated with the maximum load to fracture of the specimens. A radar graph correlates the surface energy with the load to fracture.

4. Results

Mechanical Tests

The mean shear stresses (MPa) calculated for each group are listed in Table 2. RES (69.10 ± 24.58 MPa) > Y-ZT (18.48 ± 12.12 MPa) > ENA (18.38 ± 8.51 MPa) > SUP (11.44 ± 4.04 MPa). The SUP (n = 1) and Y-ZT (n = 2) groups showed pre-test adhesive failure.

Table 2. Shear strength by mean and standard deviation in Newtons and MPa.

Groups		Failure Load			Shear Stress	
		n	Mean (N)	SD (N)	Mean (MPa)	SD (MPa)
Resin-matrix Composite		5	843.07	299.82	69.10	24.58
Rely X Ultimate	VITA Enamic	6	224.27	103.82	18.38	8.51
	VITA Suprinity	5	139.56	48.99	11.44	4.02
	VITA Y-ZT	5	225.40	147.88	18.48	12.12

SD, standard deviation; N, newtons; MPa, megapascals.

Figure 11 shows the behavior of the samples under load, and Figure 12 shows the forest plot of the shear stress by restorative material.

Despite the specimens being assembled using the same procedure, the SUP and Y-ZT groups exhibited inconsistent behavior before and during loading. The adhesion strength was material dependent. Surprisingly, the RES group performed the best, reaching mean values more than four times higher than those of the second-best ENA group.

As shown in Figures 13 and 14, the observation of the fractured specimens reveals the different mechanical behaviors of the assessed ceramics. From the observations in Table 3, failed adhesion is the unique failure mode for the Y-ZT group. The unique failure mode was cohesive in the RES group, either in the cylinder or the base, and sometimes simultaneously. Figure 15 shows a comprehensive schematic of the failure mode.

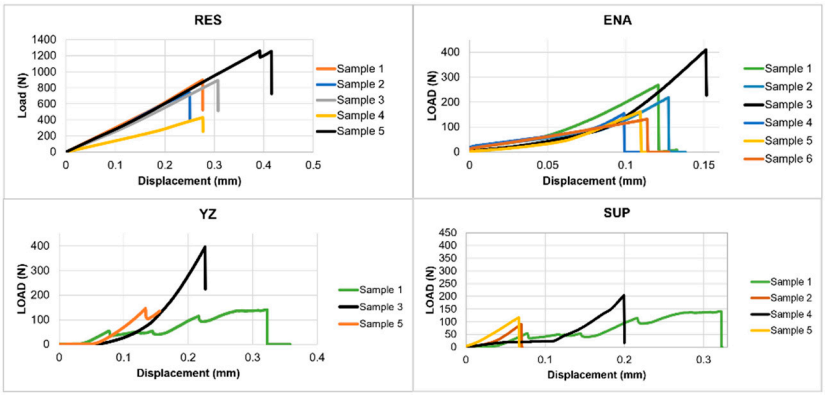


Figure 11. Graphic representation of specimen behavior under load of the control (RES), ENA, SUP, and Y-ZT groups.

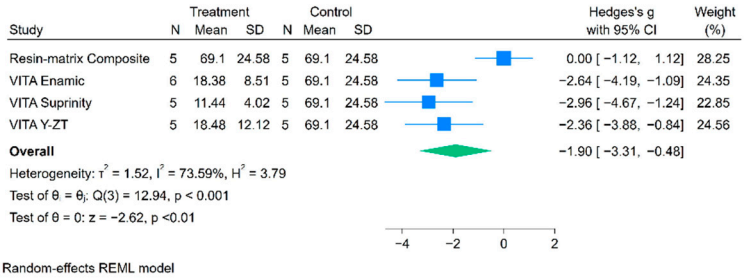


Figure 12. Forest plot of shear stress by restorative material.

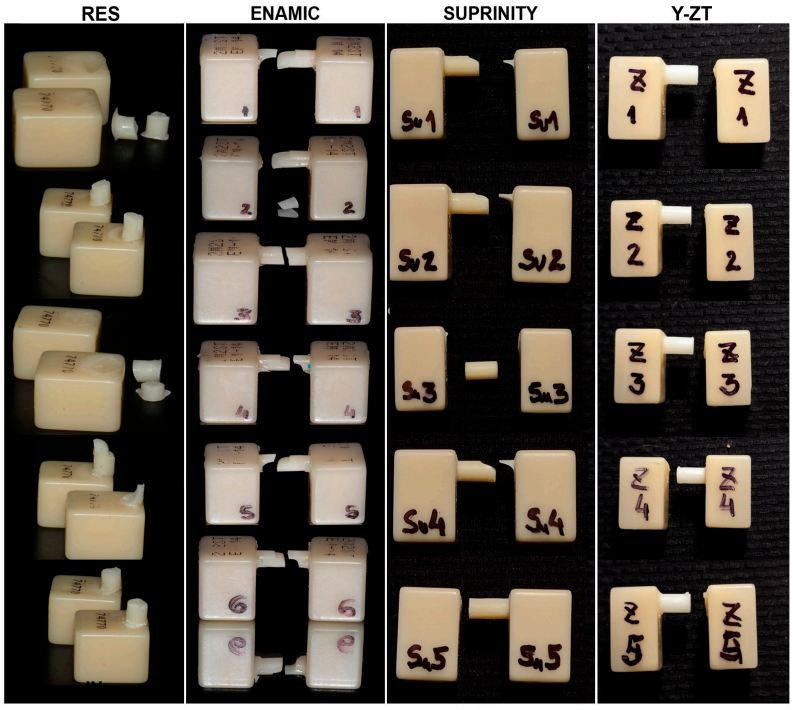


Figure 13. Fractured specimens after loading to fracture by shear forces grouped by type of material.

Table 3. Mode of failure observed in adhesive interfaces between the block and cylinder.

GROUP	Type of Failure							
	Interface 1		Interface 2		Base 1	Base 2	Cylinder	MIX
	AD	C	AD	C	C	C	C	
VITA Enamic	x						x	x
VITA Enamic							x	
VITA Enamic							x	
VITA Enamic							x	
VITA Enamic	x						x	x
VITA Enamic							x	
	Interface 1		Interface 2		Base 1	Base 2	Cylinder	
	AD	C	AD	C	C	C	C	
VITA Suprinity			x				x	x
VITA Suprinity			x				x	x
VITA Suprinity	x		x					
VITA Suprinity			x				x	x
VITA Suprinity	x							
	Interface 1		Interface 2		Base 1	Base 2	Cylinder	
	AD	C	AD	C	C	C	C	
VITA Y-ZT			x					
VITA Y-ZT			x					
VITA Y-ZT			x					
VITA Y-ZT	x							
VITA Y-ZT			x					
	Interface 1		Interface 2		Base 1	Base 2	Cylinder	
	AD	C	AD	C	C	C	C	
Nanohybrid Resin					x	x	x	
Nanohybrid Resin							x	
Nanohybrid Resin					x	x	x	
Nanohybrid Resin							x	
Nanohybrid Resin							x	

AD, adhesive failure; C, cohesive; Mix, mixed failures.

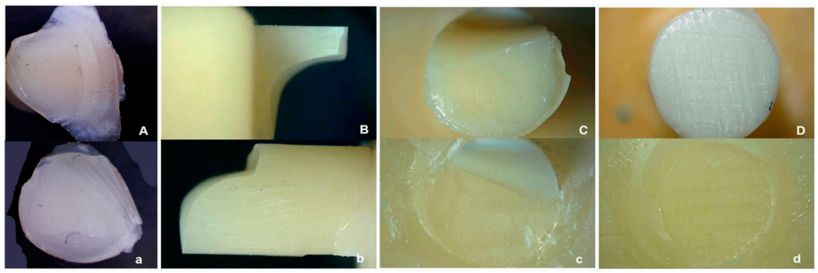


Figure 14. Examples of the mode of failure. (**A**,**a**) cohesive in the base adherend (RES_1); (**B**,**b**) cohesive in the cylinder (ENA_5); (**C**,**c**) mixed (SUP_4); (**D**,**d**) adhesive (Y-ZT_2).

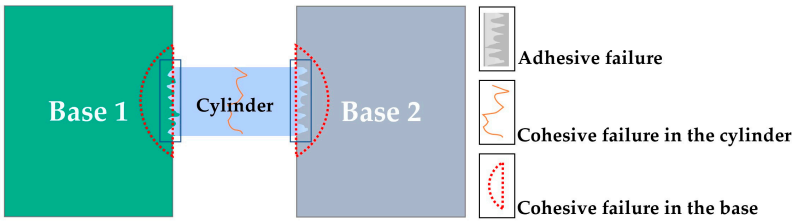


Figure 15. Comprehensive scheme of the mode of failure.

From the data obtained from the failure mechanism and surface energy of the different materials evaluated (Table 4 and Figure 16), no correlation was found between these parameters, indicating that the intrinsic chemical composition of the restorative material and its interaction with the coupling agent were the main factors affecting the mechanical behavior. Relative to the effect of the surface treatment on the CAD-CAM monolithic ceramics (Figure 17), the three treatments modified the surface of the ENA; the SUP was markedly altered by conditioning with HF 5% for 60 s and only slightly by sandblasting with AL_2O_3 50 µm, and Y-ZT was unaffected by HF 5%. These findings confirmed the data reported in the literature.

Table 4. Surface energy of the tested CAD-CAM monolithic ceramics.

	Only Grinding		
	ENAMIC	**SUPRINITY**	**Y-ZT**
Contact angle (°)	0.0	0.0	0.0
	45.9–41.0	21.5–21.6	37.5–38.2
	773–72.7	44.0–39.0	58.0–57.2
Surface Energy (mJ/m^2)	37.2	54.5	44.1
	HF 5% conditioning—60 s		
	ENAMIC	**SUPRINITY**	**Y-ZT**
Contact angle (°)	0.0	0.0	0.0
	23.6–22.8	0.0	50.1–48.6
	86.6–85.3	0.0	57.4–54.6
Surface Energy (mJ/m^2)	37.2	68.6	43.2
	Sandblasting AL_2O_3 50 µm		
	ENAMIC	**SUPRINITY**	**Y-ZT**
Contact angle (°)	0.0	0.0	0.0
	15.0–9.0	0.0	44.0–42.5
	60.7–55	0.0	60.0–59.0
Surface Energy (mJ/m^2)	46.9	68.6	42.4

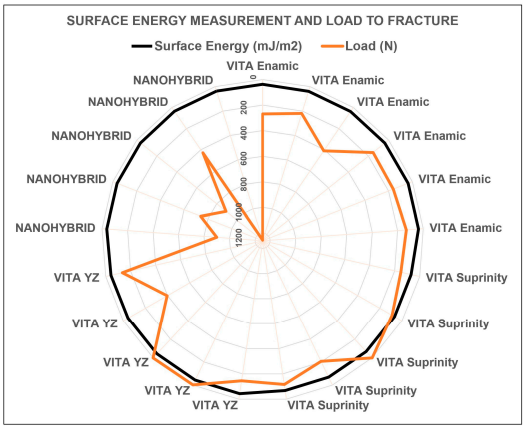


Figure 16. Radar graphic with compared mechanical performance related to the highest measured surface energy by type of CAD-CAM monolithic ceramic.

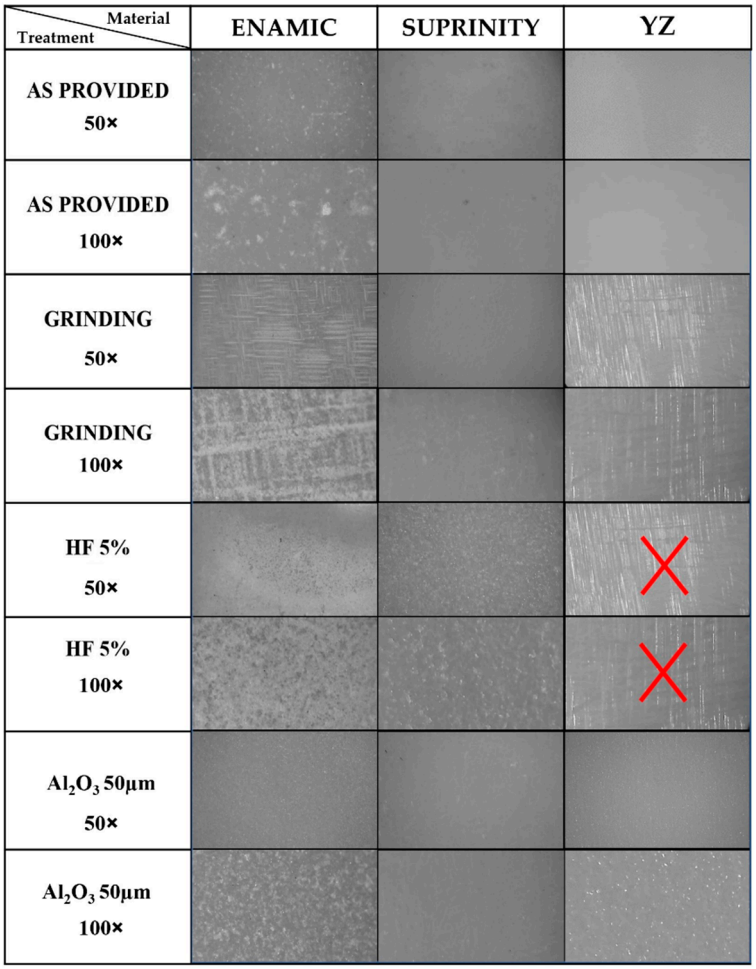


Figure 17. Microscopy observation (50× and 100× ampliation) of the CAD-CAM ceramics after different surface treatments as provided by the manufacturer, ground by coarse disk, 5% hydrofluoric acid for 60 s (HF 5%), aluminum oxide blasting (Al_2O_3 50 µm). The red cross identifies a null effect.

The crossing of microscopy and surface energy data shows that HF 5% is a suitable treatment to prepare the surface of SUP for adhesion if we only consider the microscopic interlocking between the restorative material and adhesive cement. Other materials depend on chemical reactions.

5. Discussion

The main objective of this study was to evaluate the possibility of using a standardized artificial material as a base adherent for the shear bond strength tests of restorative materials. Taking advantage of this objective and because the behavior of this material (ENA) for this purpose was unknown, CAD-CAM ceramics, from which different performances in shear bond strength testing were expected, were tested in parallel to validate the mechanical behavior of the adherend. Based on the results, the null hypothesis that the Vita Enamic hybrid ceramic was not mechanically a suitable adherend for shear bond tests was rejected, as this material withstood load forces up to 1142.89 N corresponding to an adhesive stress of 103.00 MPa. The other null hypothesis, that no differences would be found in the mechanical behavior between the CAD-CAM monolithic ceramics, was also rejected, as significant differences were found ($p < 0.01$). The meta-analysis conducted relative to different materials revealed substantial heterogeneity of results across groups due to heterogeneity rather than sampling error, with an $I^2 = 73.59\%$ of total variation and a $H^2 = 3.79$ variance between studies ($p < 0.01$).

The number of specimens used in this study reflects the surface irregularities due to manufacturing, as more than half of the cylinders were not considered suitable after 10x magnification visual inspection despite the previous calibration of the milling equipment. If the purpose is to manufacture an authentic restoration, this issue should be assessed carefully [53,54] and visual inspection with magnification before delivery should be encouraged. Nevertheless, it was not considered a real constraint for the experimental purpose, as the adherend was a homogeneous industrial material subjected to standardized surface treatment for every experimental condition, with an expected good adhesive if combined with Rely X Ultimate cement [46]. In contrast, the materials used in the cylinders were expected to differ according to the manufacturer's datasheets [25,55,56].

In this experimental design, a silicone mold was used for stabilization and standardization during the assembly. The base in PLA, because of its physical characteristics [51], ensured stabilization during mechanical tests and no flexion of the specimen, although the specimen was expected to be stable when standing alone. A silane primer was applied to the adherend surface to enhance adhesion by a chemical reaction with the polar component of the ENA structure [50,57].

Rely X Ultimate was selected based on literature [36] and parallel research [58]. The cement was used in a mixed-cure protocol (light-cure for 30 s, followed by self-cure for 10 min). To test the adhesive performance of cement was not an objective of this study. However, a control group photoactivated for only 2–3 s and left in a chemical cure for 6 min (self-adhesive mode) would be interesting to highlight the influence of chemical interactions on the success of the adhesive interface according to the cylinder material, despite the fact that the performance of this type of cement is enhanced by photopolymerization [36].

Vita Enamic was the most accessible material to handle. Vita Suprinity was very brittle in both the pre-sintered and sintered states. Polycrystalline zirconia (Vita Y-ZT) was accessible for milling, but it was almost impossible to separate the cylinders after the block had been sintered, with the destruction of several diamond points in the process. In future studies, we recommend separating cylinders before sintering. Resin-matrix composites (RES) are easy to handle; however, the possibility of including air bubbles in the cylinder upon production was a concern. Given the unexpected performance of the RES cylinder, not having determined the ultimate strength of this material is a limitation of this study, because it would have been interesting to compare it with data relative to the other materials used.

The correlation between adhesive stress and failure mode confirmed that the limitation of experimental Y-ZT RBBs lies in the success of adhesion, which agrees with the results of previous studies [32,59]. In fact, despite being the toughest material, the Y-ZT group, if the failed specimens are excluded, achieved mechanical performance similar to the ENA group (18.48 $\pm$ 12.12 MPa and 18.38 $\pm$ 8.51 MPa, respectively), which has a toughness about 8 times lower [55,56]. The exclusive adhesive failure in the Y-ZT group, including pre-test failures, reinforces the need for an easily replicable and efficient adhesion protocol when working with this type of material [1], especially in the case of a minimally invasive one-retainer anterior RBB, which does not have additional macromechanical retention [32,59].

Despite several searches of the literature and thousands of articles found related to adhesion, namely relative to zirconia [60], no studies were found that would allow for a comparison of the results of this study with other existing ones. This is due to the lack of comparable adhesive protocols or adherents, which agrees with a recent meta-analysis that identified 686 protocols to adhere 37 different CAD-CAM blocks [1], but also with the fact that, frequently, results are not available in MPa for evidence-based comparisons. Some studies have tested this type of CAD-CAM ceramic or adhesive cement. However, these studies did not use them simultaneously, nor did they evaluate them with a similar experimental setting to that of the current study, as CAD-CAM ceramics are often tested in the form of a one-piece fixed crown subjected to catastrophic fracture or pull-out tests. Other studies have evaluated CAD-CAM ceramics adhered to a cement cylinder or as a block adhered to another block.

This limitation reinforces the importance of this study because the experimental setting approaches a clinical situation of a minimal invasive resin-bonded bridge. It also showed that an industrial material could be used, at least in preliminary tests, as an adherend in shear strength tests of CAD-CAM restorative materials. This type of adherend allows for standardization and overcomes the existing constraints found in the use of biological substrates.

6. Recommendations for Future Research

Considering the potential of the tested adherend, experimental models to evaluate the shear resistance of cement with different adhesive strategies are recommended.

With CAD-CAM materials in rapid evolution, namely, those produced by addition, the use of this type of adherent could facilitate a quick and standardized evaluation of their adhesive strength, allowing easy comparison with existing CAD-CAD monolithic materials, for which there is already some scientific evidence.

7. Conclusions

The VITA Enamic block is a potential base adherent for SBS tests because it resists a shear load of up to 103 MPa (RES sample 5 test) in a cylinder with a double-interface connection design. Significant differences in the mechanical behavior with respect to the shear strength were identified between the tested CAD-CAM ceramics. Under the experimental conditions of this study, the SBS was not related to the surface energy of the substrates, and the failure mode was material-dependent. As a restorative material, ENA is predictable and easy to handle. The SUP was difficult to handle owing to its brittleness in both the pre-sintered and sintered states. The Y-ZT failure mode was always adhesive.

The tested CAD-CAM ceramics have sufficient adhesive strength to be used as resin-bonded bridges for permanent or interim rehabilitation, provided an efficient adhesive protocol is wisely chosen, and the need for short-term removal, equated as Y-ZT, is very difficult to remove by drilling.

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