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Shear Bond Strength and Failure Mode of a Novel Experimental Short Fiber-Reinforced CAD/CAM Block Material (In Vitro Study)

Keywords
CAD/CAM block, Resin-based composite, Short-fiber-reinforced composite, Shear-bond strength test, bond strength.

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Abstract
Although short fiber-reinforced composites demonstrate favorable mechanical properties, their bonding performance as CAD/CAM materials is not well documented specifically for indirect restoration. This study compared the shear bond strength of a novel experimental SFRC CAD/CAM block with conventional resin-based CAD/CAM materials with different microstructures. Field-emission scanning electron microscopy (FESEM) was used to examine the microstructural features of specimens from each group. For shear bond strength testing, specimens were allocated into three groups: Group 1 (Grandio Blocs), Group 2 (Cerasmart 270), and Group 3 (experimental SFRC CAD/CAM block), with 15 specimens in each group. After surface treatment by air abrasion with aluminum oxide (50 µm particles at 2 bar pressure), the primer was applied, and a dual-cured self-adhesive resin cement was used to bond all specimens. Subsequently, a universal testing machine equipped with a 5 kN load cell was used to debond the specimens. A FESEM analysis revealed that the experimental SFRC CAD/CAM block consisted of a polymeric resin matrix reinforced with randomly oriented and uniformly dispersed short fibers. In contrast, Grandio Blocs exhibited a densely packed nanohybrid microstructure characterized by irregularly shaped inorganic fillers. Cerasmart 270 demonstrated a more homogeneous microstructure, with evenly distributed nanosized spherical fillers within the resin matrix. The Experimental SFRC showed the highest bond strength (17.279 ± 0.711 MPa), surpassing the Grandio blocs (12.607 ± 0.781 MPa) and Cerasmart 270 groups (12.703 ± 0.593 MPA) ($p < 0.001$) and highlighting its superior resistance to debonding. The Grandio blocs and Cerasmart 270 groups were not statistically different from each other ($p = 1.000$). The presence and distribution of reinforcing fibers in the experimental block likely account for its higher shear bond strength compared with other groups that provide more microretentive sites for silane coupling.

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1. INTRODUCTION

Advances in digital dentistry have significantly influenced the development of modern indirect restorations, particularly those fabricated using CAD/CAM systems. These restorations are typically produced by milling prefabricated ceramic or resin composite blocks [1]. Despite their widespread use, ceramic materials are characterized by intrinsic brittleness, which increases their susceptibility to crack formation and sudden fracture under occlusal loading, potentially affecting their long-term clinical reliability. In addition, the high hardness of ceramics may contribute to increased wear of opposing natural teeth [2,3]. Resin-based CAD/CAM materials, on the other hand, present several clinical and practical advantages. Their lower elastic modulus provides greater resilience and reduced brittleness, enhancing their capacity to withstand functional stresses [4]. These materials also exhibit lower hardness, which facilitates the milling process, minimizes bur wear, and decreases the likelihood of chipping or catastrophic failure during fabrication [5,6]. Furthermore, resin composites are associated with reduced abrasiveness toward antagonist dentition [7,8]. From a clinical perspective, CAD/CAM resin composites offer cost-effective fabrication due to simplified processing, the elimination of firing steps for crystallization or staining, shorter milling times, extended bur longevity, and the possibility of straightforward intraoral repair. When appropriate surface conditioning protocols are applied, these materials also demonstrate reliable bonding to resin cements [8].

Numerous strategies have been explored to enhance the mechanical performance of extensive composite restorations as well as the supporting dental hard tissues. In recent years, manufacturing advancements have led to the development of reinforced CAD/CAM composite blocks, in which improvements in filler technology have been implemented to achieve superior mechanical characteristics [4]. Among these developments, particular attention has been directed toward short fiber-reinforced composites (SFRCs), which incorporate discontinuous glass fibers into the resin composite matrix. The inclusion of these fibers is designed to alter crack initiation and propagation pathways, thereby improving the fracture resistance of the material [9]. A variety of fiber types have been applied in clinical dentistry; however, S-glass and E-glass fibers are the most widely used due to their ability to form effective silane-mediated bonding with the resin matrix [10]. Achieving a strong and durable bond between indirect CAD/CAM restorations and resin cement is vital for the long-term success of treatment [11]. Surface treatments, whether mechanical by air-particle abrasion or chemical by acid etching, that create greater surface irregularity can effectively enhance this bond. These methods improve the bond strength between CAD/CAM restorations and resin cement by creating micro-mechanical retention [12,13]. When the surface area of restoration is increased, it creates more sites for micromechanical interlocking and exposes a greater number of fillers that can chemically bond through silane. This improves both the mechanical retention and chemical adhesion at the resin-restoration interface. Conversely, materials with smoother, highly cross-licked, or densely packed matrix expose fewer reactive sites after surface treatment and decrease the binding strength. Therefore, the surface topography and composition of different restorations directly influence the durability of these restorations [12]. The performance and reliability of CAD/CAM composite restorations can be assessed through a range of laboratory parameters, encompassing mechanical, bonding, and esthetic properties [14]. Among the various bond evaluation methods, the shear bond strength (SBS) test has gained widespread acceptance due to its methodological simplicity, standardized testing protocol, and ability to produce rapid and reproducible results [15]. This test holds particular relevance because it replicates shear stresses, which represent one of the primary factors contributing to bonding failure of restorative materials under intraoral conditions [16].

Previous investigations have demonstrated that short fiber-reinforced composites exhibit favorable mechanical performance, including enhanced fracture resistance and acceptable surface characteristics when used for indirect restorations. Nevertheless, limited information is available regarding the bonding performance of SFRC materials specifically developed for CAD/CAM-based indirect applications. Therefore, the present study aimed to assess the shear bond strength of a novel experimental SFRC CAD/CAM block and to compare its performance with that of conventional resin-based CAD/CAM materials with different microstructures.

2. MATERIALS AND METHODS

2.1. Study design and testing materials

Table 1 summarizes the three resin-based CAD/CAM composite blocks evaluated in this study. The materials were selected based on differences in their microstructural characteristics. Specimens were allocated into three experimental groups: Group 1—Grandio Blocs, Group 2—Cerasmart 270, and Group 3—experimental SFRC CAD/CAM block, with fifteen specimens assigned to each group.

Table 1: Tested materials

CAD/CAM block	Type	Manufacturer	Matrix	Filler
Cerasmart 270	Dispersed fillers resin-based composite	GC Crop., Tokyo, Japan	Bis-MEPP UDMA DMA	Silica (20nm), barium glass (300 nm) Wt 71%
Grandio blocs	Dispersed fillers resin-based composite	VOCO, GmbH, Cuhhaven, Germany	UDMA DMA	Silica 20-60 nm) 86 wt %, barium-alumino-borosilicate
Experimental SFRC block	Short fiber reinforced composite (SFRC)	Gc, Tokyo, Japan	Bis-MEPP, TEGDMA, UDMA,	Micrometer scale E-glass fibers 25 wt%, barium glass fillers 75 wt% %

2.2. The Novel Experimental SFRC CAD/CAM block

The experimental short fiber-reinforced composite (SFRC) CAD/CAM block used in this study was prepared and previously characterized in detail in an earlier publication by the authors [17]. Therefore, only a concise description of the material composition is provided here. The experimental SFRC block consisted of discontinuous E-glass microfibers with a diameter of approximately 6 µm and a length ranging from 200 to 300 µm. The microfibers were identical to those used in the commercial flowable SFRC material (everX Flow; GC Corporation, Tokyo, Japan). The composite formulation comprised a resin matrix (30 wt%), silanated inorganic particulate fillers (45 wt%), and randomly oriented glass microfibers (25 wt%) [17]. The experimental SFRC material was consolidated into a CAD/CAM-milled block using compression and heat-curing under controlled conditions, as described in the previous study [17]. The fabricated blocks were then used for specimen preparation and subsequent shear bond strength testing in the present investigation, and compared with two commercially available resin-based CAD/CAM blocks with different microstructural composition—Cerasmart 270 (GC Corporation, Tokyo, Japan) and Grandio Blocs (VOCO GmbH, Cuxhaven, Germany).

2.3. Specimen preparation

Disc-shaped specimens were prepared for shear bond strength testing using computer-aided design software (Chitubox, version 1.9.5). The STL files were transferred to a CAD/CAM milling unit (CORiTEC 350 i PRO; imes-icore GmbH, Eiterfeld, Germany), and specimens were fabricated under wet grinding conditions following the manufacturer's instructions. After milling, all specimens were ultrasonically cleaned in distilled water for 1 min, air-dried, and inspected under a stereomicroscope to detect surface defects. Dimensional accuracy was verified using a digital caliper to ensure consistency among samples. Field Emission Scanning Electron Microscopy (FESEM; Inspect F50, FEI) analysis was performed to examine the microstructural features of specimens from each group following surface polishing with diamond paste.

2.4. Bond strength test

2.4.1. Embedding of the specimen in the specimen holder

Fifteen milled disc-shaped specimens, each with a diameter of 15 mm and a thickness of 2 mm, were mounted in cylindrical acrylic blocks as a base, which are suitable for use with a universal testing machine. The custom-made cylindrical silicon was fabricated, which has a height of 30 mm, an internal diameter of 24 mm, and a circular depression in its center with the same diameter (10 mm) as the milled specimens. The depression was 0.2 mm; this will enhance centralizing the specimens and project them out of the acrylic block by 0.2 mm [18]. All specimens were held and positioned in the circular depression using a dental tweezer. Then, a self-cured acrylic resin was mixed and poured into the mold. Specimens in all groups were color-coded blue for Grandio Bloc, red for Cerasmart 270, and green for the experimental SFRC to identify each group (Fig. 1). After the completion of polymerization, the acrylic mold was removed from the silicon mold, with the specimen embedded in it (Fig. 2).

The surface of the embedded specimens was air-abraded with aluminum oxide particles (50 µm, pressure 2 bar) for 15 seconds. Then, all specimens were washed and air-dried before the application of the primer (BIS-SILANE A & B, from BISCO Dental Products, Schaumburg, IL, USA). After the application of aluminum oxide particles, specimens from each group were prepared for SEM study to assess the effect of airborne particles on the surfaces of each material.

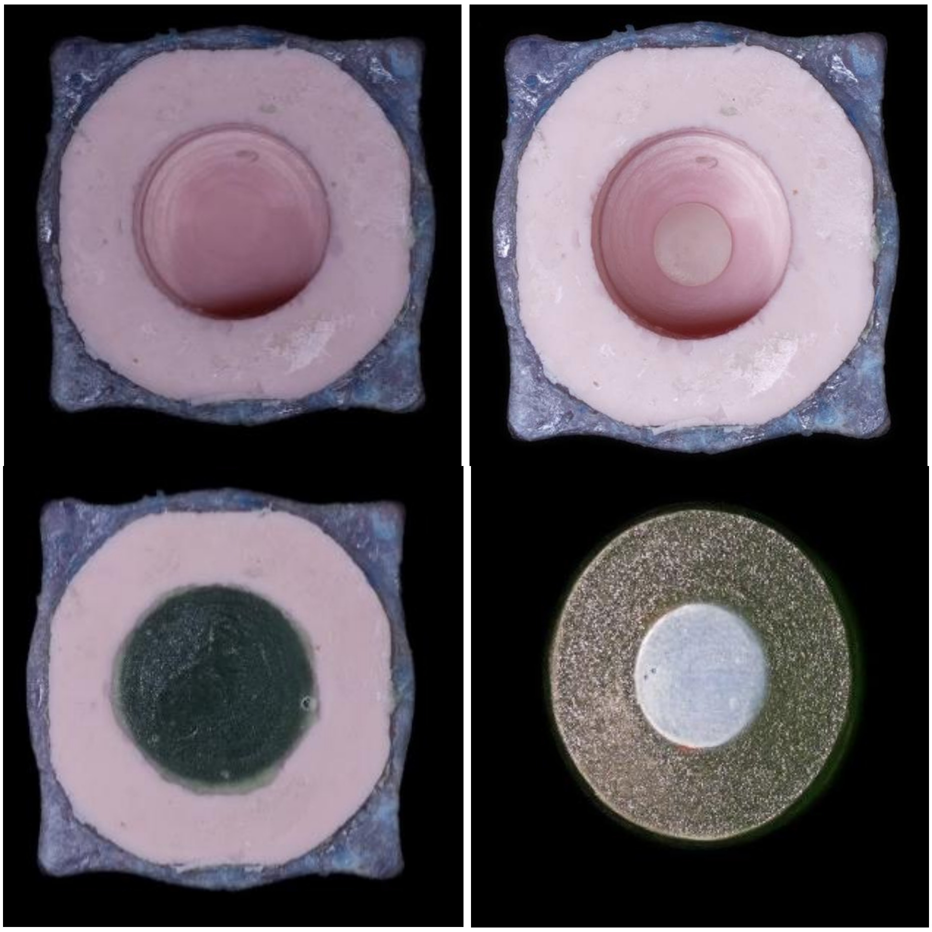

Fig.1 Steps of embedding the specimen in the specimen holder.

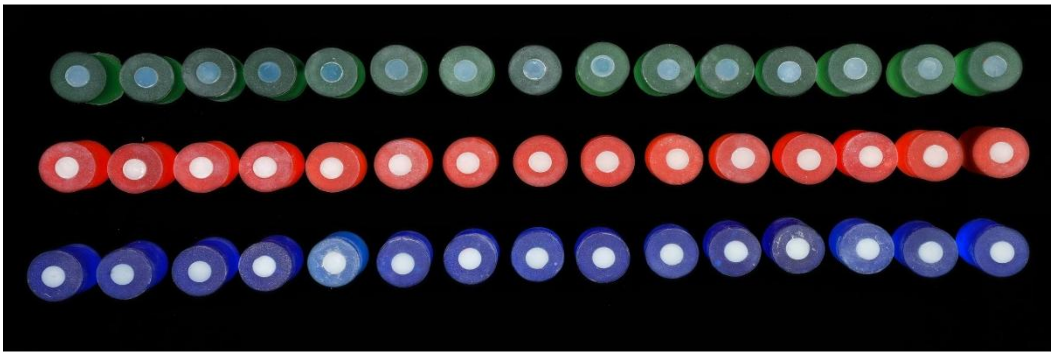

Fig.2 Specimens from different groups were mounted in an acrylic holder for bond strength testing

2.4.2. Application of resin cement

A Teflon split mold was fabricated for applying resin cement to milled specimens of each group. The metal clamps were used to hold and position the Teflon mold on the acrylic block, which held the specimen. The inner part of the Teflon mold was flat, which fitted firmly over the acrylic mold when the metal clamps were locked. The external part of the Teflon mold featured a small cylindrical opening in its center, measuring 5 mm in diameter and 2 mm in depth, for the application of resin cement. Two holes were created on each surface of the split mold to enhance the separation of resin cement without dislodgment. The Teflon split mold was assembled on top of the acrylic block, which held the specimen, with the aid of clamps. The dual-cured self-adhesive resin cement (TheraCem, BISCO Dental Products, Schaumburg, IL, USA) was applied through the mixing tip into the split mold in the center of the surface-treated specimens in all groups (Fig.3). The excess cement was removed. The specimens were light-cured from the top for 20 seconds using an LED light-cure device at a light intensity of 1470 mW/cm² (Eliper DeepCure-L, 3M ESPE, USA). Then, the thumb nuts were loosened, and the Teflon mold was carefully disassembled. The specimens were then light-cured again on each side. The excess flash from the base of the resin composite was removed with a blade. Finally, all specimens were stored in distilled water at 37 °C.

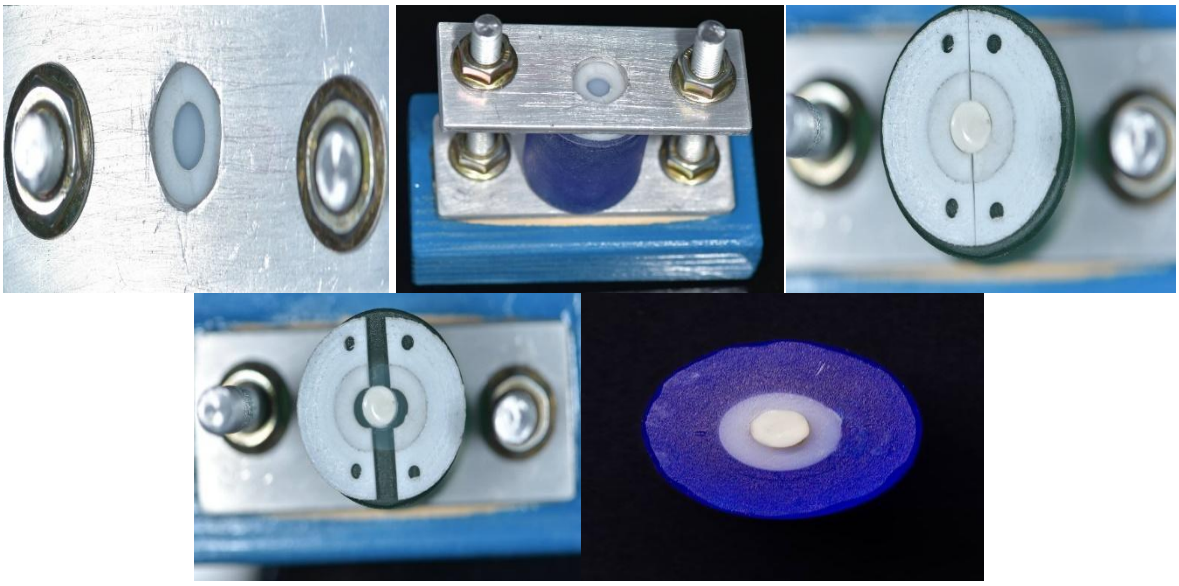

Fig.3 Custom-made split mold for application of resin cement

2.4.3. Application of Shear Force

The shear bond strength test was used to identify the quality of adhesion between the luting cement and the composite materials; fifteen specimens were selected from each group for the shear bond strength test. A universal testing machine (GOTECH, Taichung City, Taiwan) with a 5 KN load cell was used to debond all specimens. The acrylic blocks holding the samples were secured in a custom-made holder for mounting in the jig of the universal testing machine. The chisel was used to apply force to all samples during the testing procedure. The shear force was applied parallel to the interface between the resin cement and embedded specimen at a cross speed of 1 mm/min until failure was registered (Fig.4). Data was registered by (U-27 GOTECH) software, and the maximum load at failure (N) was divided by the bonding area (mm2) to calculate the bond strength in megapascal (MPa).

Fig.4 The specimen during the shear bond strength test

2.4.4. Failure mode analysis

The modes of failure were analyzed with a stereomicroscope (TX-350s, Kaisi, China) at 40X magnification, and they were classified either as cohesive failure within CAD/CAM resin-based material or adhesive failure between CAD/CAM resin-based material and resin luting cement or mixed failure. The effect of surface treatment with air particle abrasion on the CAD/CAM resin-based substrate of each group was observed using Field Emission Scanning Electron Microscopy (FESEM) Inspect F50 FEI (Fig. 7).

3. RESULT

3.1. SEM characterization

SEM images revealed that the experimental SFRC CAD/CAM block consists of a dual-phase composite, comprising a polymeric resin matrix reinforced with short fibers. The fibers were distributed uniformly throughout the matrix and oriented randomly in multiple directions, indicating effective dispersion during fabrication. The images showed that the fibers were well embedded within the resin, suggesting intimate fiber-matrix contact essential for efficient stress transfer and material durability (Fig.5 A,B). The fibers measured approximately 7 µm in diameter and exceeded 300 µm in length, consistent with typical short fiber reinforcements used in dental composites. Additionally, the presence of nanoparticles, likely nano-silica or alumina, with grain sizes below 100 nm, was observed within the matrix.

Distinct microstructural characteristics were also identified in the two commercial CAD/CAM blocks.

Grandio Blocs displayed a densely packed nanohybrid structure, with irregular, closely clustered inorganic fillers embedded within the resin, resulting in a compact, granular morphology with minimal inter-filler spacing (Fig.5 C). In contrast, Cerasmart 270 exhibited a more homogeneous microstructure, characterized by uniformly dispersed nanosized spherical fillers within the resin matrix, producing a smoother and more uniform surface (Fig.5 D).

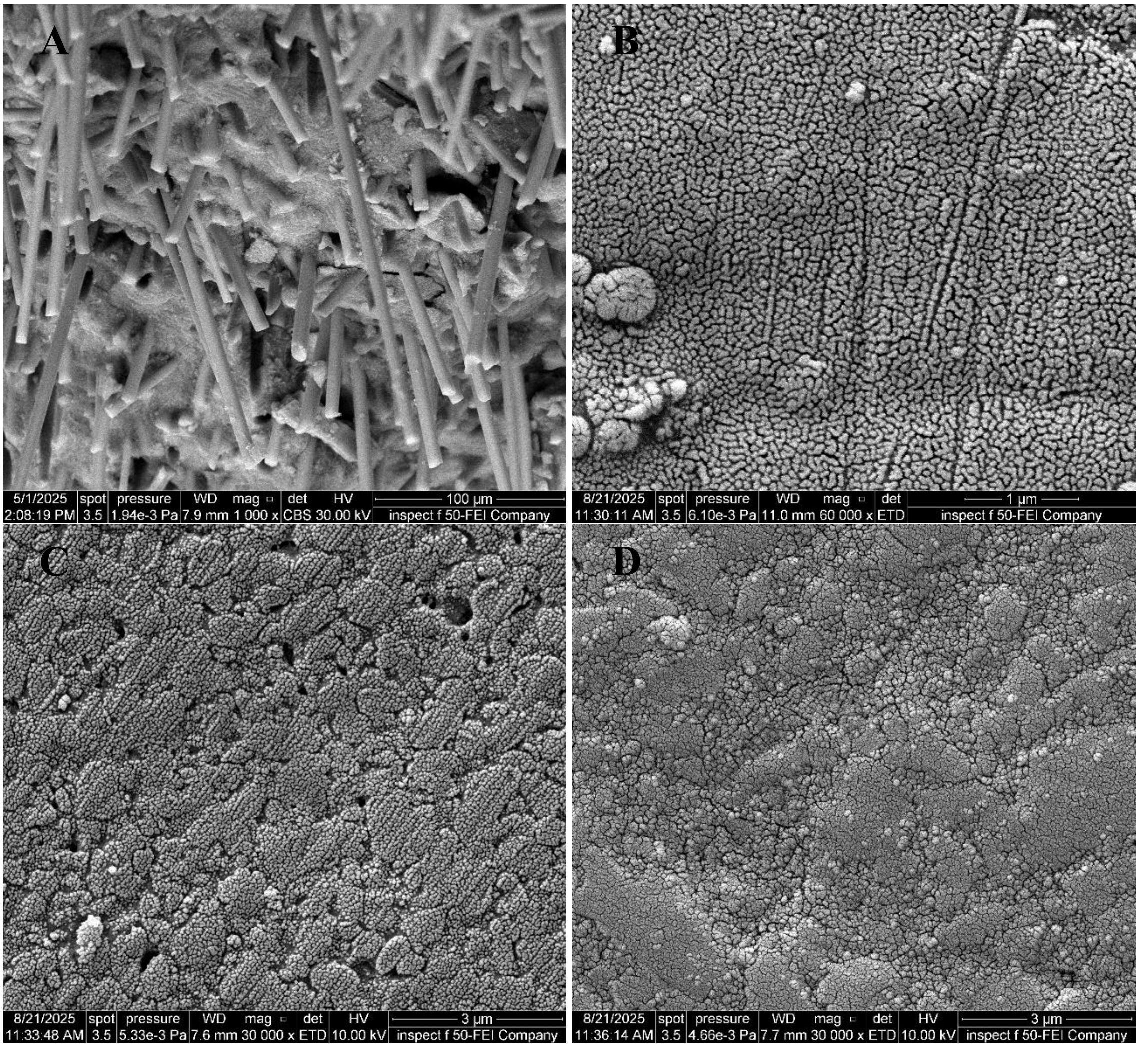

Fig. 5: SEM images of the CAD/CAM resin-based blocks. (A) Experimental SFRC specimen at 1000× magnification, illustrating the distribution of short fibers within the resin matrix. (B) Experimental SFRC, (C) Grandio Blocs, and (D) Cerasmart 270 at 30,000× magnification, highlighting the densely packed nanofillers in Grandio Blocs and the more uniform filler distribution in Cerasmart 270.

3.2. Bond strength test

Shear bond strength refers to the stress required to cause sliding failure at the bonded interface, with higher values indicating stronger adhesion and improved resistance to debonding. Statistical analysis revealed a highly significant difference in shear bond strength among the tested groups (one-way ANOVA; $F(2, 42) = 218.821$, $p < 0.001$), as presented in Table 2,3. The experimental SFRC CAD/CAM block exhibited the highest mean shear bond strength (17.279 ± 0.711 MPa), which was significantly greater than that of both Grandio Blocs and Cerasmart 270 ($p < 0.001$). No statistically significant difference was observed between the Grandio Blocs and Cerasmart 270 groups ($p = 1.000$).

Table (2) Descriptive statistic presentation of bond strength test (MPa)

Group	**N**	**Mean ± SD**	**95% CI (Lower-Upper)**	
Grandio blocs	15	12.607 ± 0.781	12.174 - 13.039	
Cerasmart 270	15	12.703 ± 0.593	12.375 - 13.032	
Experimental SFRC	15	17.279 ± 0.711	16.886 - 17-673	

Table 3: Pairwise mean differences, confidence intervals, and effect sizes for Shear Bon Strength

Tests	Groups		Mean Difference (I-J)	SE	P-Value	95% Confidence Interval		Effect Size
						Lower Bound	Upper Bound	
Shear Bond strength (MPa)	Grandio blocs	Cerasmart 270	-0.097	0.255	1.000	-0.733	0.54	0.139
		Experimental SFRC	-4.673	0.255	0.000	-5.309	-4.036	6.258
	Cerasmart 270	Experimental SFRC	-4.576	0.255	0.000	-5.213	-3.939	6.993

Table 4 summarizes the failure modes observed for each CAD/CAM resin-based material following shear bond strength testing. The experimental SFRC group predominantly exhibited adhesive failures. In the Grandio Blocs group, failures were mainly mixed, with adhesive failures also frequently observed. In contrast, Cerasmart 270 demonstrated exclusively cohesive failures across all specimens.

Table (4) The distribution of the mode of failure according to different CAD/CAM resin-based substrates after aluminum oxide abrasion

Groups	Adhesive failure	Cohesive failure	Mixed failure
Grandio blocs	7	0	8
Cerasmart 270	0	15	0
Experimental SFRC	15	0	0

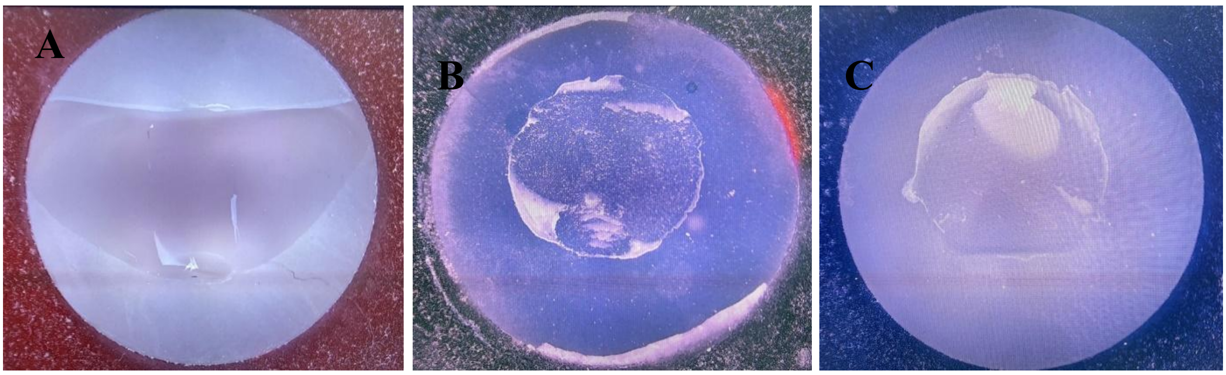

Fig.6 A.Cohesive failure, B.Adhesive failure, C. Mixed failure

4. DISCUSSION

With the introduction of computerised technology, CAD/CAM resin composite materials have become available in the dental market. Because CAD/CAM resin composite materials are manufactured by compressing and heat-curing in a standardized environment, their mechanical properties show significant improvement compared to conventionally produced resin composite materials. This is due to a higher conventional rate in CAD/CAM composite compared to conventional composite [19]. The improved mechanical properties of resin-based composites are generally attributed to modified microstructural features, including the polymer matrix, type and loading of fillers, and the quality of the filler-matrix interface [20].

The present study aimed to assess the shear bond strength of an experimental short fiber-reinforced composite (SFRC) CAD/CAM block in comparison with two commercially available resin-based CAD/CAM materials, Grandio Blocs and Cerasmart 270, which exhibit different microstructural characteristics. Cerasmart 270 contains approximately 71 wt% silica and barium glass nanoparticles, whereas Grandio Blocs is characterized by a higher filler loading of about 86 wt%. SFRC materials are composed of a reinforcing phase in the form of fibers embedded within a continuous resin matrix, creating a multiphase composite system [21]. In the present study, the experimental SFRC block incorporated discontinuous E-glass microfibers with a diameter of approximately 6 µm and lengths ranging from 200 to 300 µm, corresponding to the fibers used in the commercial flowable SFRC material everX Flow (GC Corporation, Tokyo, Japan). The composite formulation consisted of a resin matrix (30 wt%), silanated inorganic particulate fillers (45 wt%), and randomly oriented glass microfibers (25 wt%) [22]. Key fiber-related parameters, including aspect ratio, critical fiber length, fiber content, and fiber orientation, are known to influence the mechanical behavior of SFRC materials [23]. The aspect ratio, defined as the ratio of fiber length to fiber diameter, plays a critical role in reinforcement efficiency. In this study, the microfibers exhibited an aspect ratio exceeding 30, which has been reported as the minimum threshold required to achieve effective reinforcement in short fiber-reinforced composites [24].

Numerous studies have focused on enhancing the bonding quality of resin-based CAD/CAM materials when tested with various luting resin cements [25,26,27]. In the current study, Self-adhesive resin cement was used for cementing all specimens as it is widely and easily used in daily practice [28]. According to previous studies by Mine et al. (2019) and Şişmanoğlu and Turunç-oğuzman (2020), it has been recommended that micro-retentive surfaces be created before application of resin cement by either etching with hydrofluoric acid or airborne-particle abrasion for resin-based CAD/CAM blocks [29,30]. According to the literature, airborne-particle abrasion with 50 µm Al_2O_3 and a pressure of 0.1-0.2 MPa yielded better bond strength values [25,30]. A study by Tekçe et al. (2018) showed that the application of airborne-particle abrasion for longer than 30 seconds decreases the bond strength of resin-based CAD/CAM materials [31]. Therefore, in this study, all surfaces of each group were treated with 50 µm Al_2O_3 airborne-particle abrasion at 2 bar for 15 seconds based on these recommendations. Silane application was used, as it is generally believed that silane improves the bond strength values of resin-based materials by generating micro-retentive surfaces [32,33]. In the literature, the micro-tensile and shear bond tests are the most widely used techniques for calculating bond strength values, and their pros and cons have been extensively discussed in the past. Compared to the micro-tensile test, the shear bond test is considered one of the rapid and convenient techniques for obtaining authentic results. In vitro SBS calculation has been noted to align well with clinical practice bond performance [16]. For this reason, the shear bond test was employed in the present research to investigate the effect of various resin-based CAD/CAM blocks with different compositions on bond strength performance. Based on our results, the experimental SFRC group exhibited a higher SBS value than the Grandio blocs and Cerasmart 270 groups, with no significant difference observed between the Grandio blocs and Cerasmart 270 groups. The same finding was observed by Fouquet et al. (2022), who reported no significant difference in bond strength between Grandio blocs and Cerasmart 270 when the surface of each block was treated with sandblasting and saline [34]. The effect of airborne-particle abrasion on the treated surface of the experimental SFRC group was different from Grandio blocs and Cerasmart 270, as the effects depend on the particle size and filler composition [35]. SEM images of Grandio blocs and Cerasmart 270 showed small surface porosities, mainly in the same way, after treatment with airborne-particle abrasion. In contrast, the experimental SFRC exhibited incompletely dissolved fibers, which created surface irregularities and provided a higher surface area for bonding (Fig.7). This finding confirms that airborne-particle abrasion can have a significant effect on SFRC CAD/CAM blocks, providing precise micromechanical interlocking with resin cement that leads to long-lasting adhesion. In the experimental SFRC group, after the application of airborne-particle abrasion, the exposed fibers increase the available surface area and generate microretentive sites for silane coupling, thereby enhancing micromechanical interlocking and chemical adhesion to resin cement. In contrast, commercial CAD/CAM blocks with fully cross-linked nanohybrid or nanocluster filler matrices provide fewer exposed reactive sites after sandblasting, limiting interfacial adhesion. Therefore, the presence and distribution of reinforcing fibers in the experimental block likely account for its higher shear bond strength compared with other groups.

Notable distinctions were found concerning the failure mode of Grandio blocs, Cerasmart 270, and experimental SFRC groups; these differences may be attributed to variations in the filler compositions and resin matrix of these blocks, and to the way each material interact with airborne particle abrasion. In the experimental SFRC group, no cohesive failure was observed, whereas in the Cerasmart 270 group, all samples failed cohesively. The failure type in the Grandio blocs group was mixed and adhesive (Fig. 6). The fiber fillers in the experimental SFRC group provided the matrix with more toughness, enabling it to withstand shearing stress and prevent a cohesive fracture. The matrix of the Cerasmart 270 group could be weakened by airborne particle abrasion, potentially leading to catastrophic failure, indicating that the intrinsic material was the weakest link, which means the crack initiation and propagation occurred inside the block rather than at the bond. Sirisha et al. (2014) found that the presence of cohesive failure indicates that adhesion values exceed the material's cohesive strength. The adhesive/mixed failures for Grandio Blocs indicate that airborn particle abrasion substantially improved micromechanical retention and interfacial bonding, while some regions of the near-surface substrate weakened by abrasive treatment [36].

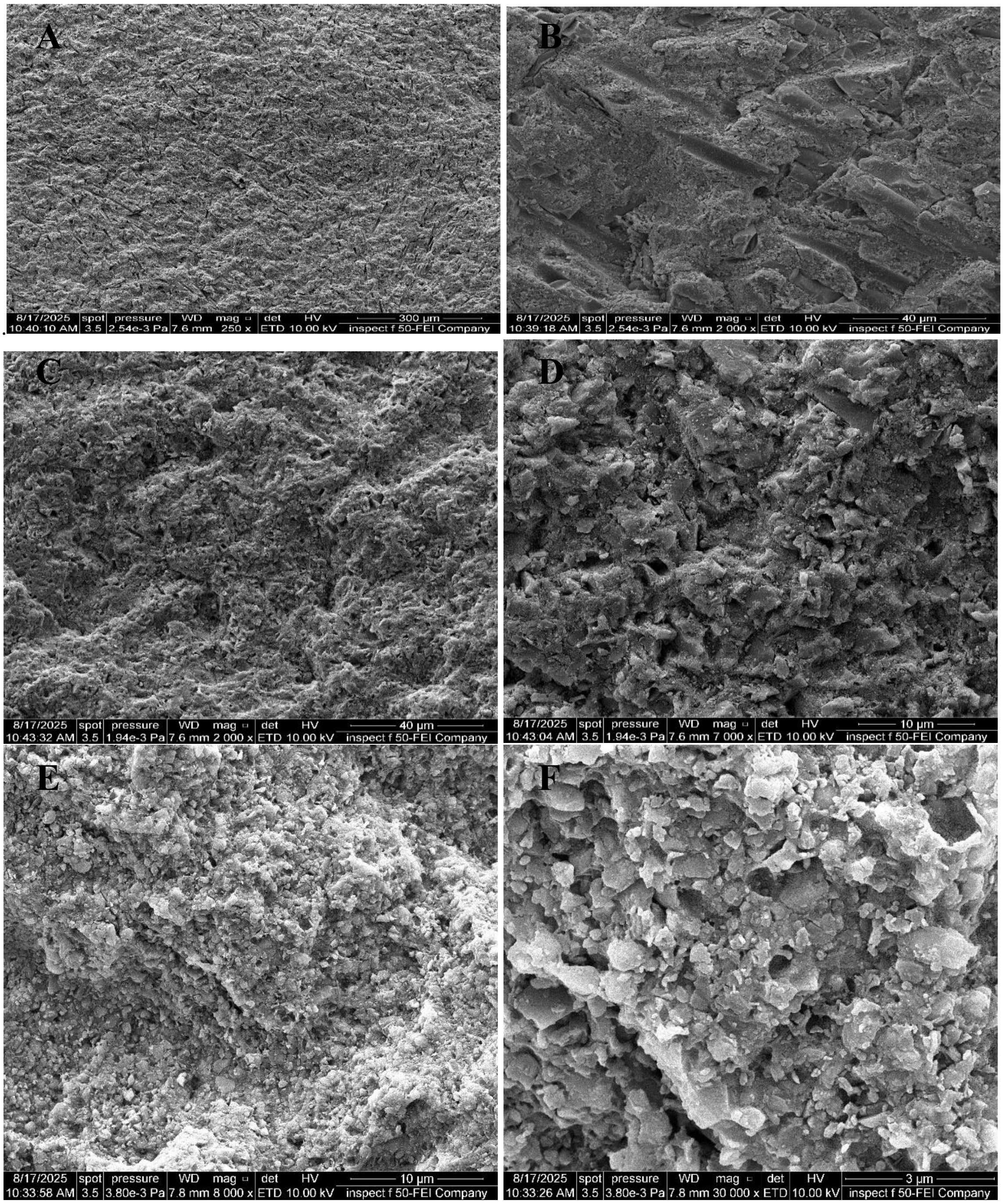

Fig.7 SEM images of specimen surfaces viewed at different magnifications after air particle abrasion. (A, B) The experimental SFRC; (C, D) Grandio blocs; (E, F) Cerasmart 270

5. CONCLUSION

The experimental SFRC CAD/CAM block exhibited significantly higher shear bond strength than both Grandio Blocs and Cerasmart 270, suggesting an improved resistance to debonding and potential benefits in clinical situations involving high functional stresses. This superior bonding performance may be attributed to the incorporation of short reinforcing fibers that are well integrated within the resin matrix and filler system. The presence and spatial distribution of these fibers likely enhance micromechanical interlocking and provide additional reactive sites for silane coupling, thereby promoting stronger interfacial adhesion. In contrast, to other CAD/CAM blocks characterized by highly cross-linked nanohybrid or nanocluster filler structures may present fewer accessible reactive sites following surface treatment, which could limit the effectiveness of bonding procedures and result in lower shear bond strength.

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