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ORIGINAL ARTICLE

Shear-bond strength and optical properties of short fiber-reinforced CAD/CAM composite blocks

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Abstract

The aim of this study was to assess the shear-bond strength (SBS) of resin-luting cement to experimental short fiber-reinforced CAD/CAM composite (SFRC) compared to conventional CAD/CAM (Cerasmart 270), 3D printed (GC TEMP PRINT, Pro3dure GR-17), and laboratory (Gradia Plus) composites. Moreover, translucency parameter values and light transmission were evaluated. For each of the five types of composites, discs were prepared (n = 16/group) and divided into subgroups (n = 8/group) according to surface treatment protocol (hydrofluoric acid or air-particle abrasion). SBS test was performed using universal testing machine until failure, and failure modes were visually analyzed. Translucency parameter and curing-light transmission values through 1, 2, and 3 mm thickness were quantified using spectrophotometry and the MARC resin calibrator, respectively. Scanning electron microscopy (SEM) was used to examine the CAD/CAM composites after surface treatment. Composite type and surface treatment had a significant effect on SBS. Laboratory composite showed the highest SBS value (22.4 MPa). Cerasmart 270 exhibited higher translucency parameter values (19.8, 11.0, 5.0) than SFRC (14.5, 5.2, 1.6) at the three composite thicknesses tested. Air-particle abrasion was more effective in enhancing SBS than acid etching. Experimental SFRC CAD/CAM composite showed higher SBS values than Cerasmart 270. For all composites, translucency parameter values and light transmission decreased as thickness increased.

KEYWORDS
3D printed composite, air-particle abrasion, laboratory composite, shear-bond strength test

INTRODUCTION

Development in digital technology in recent years has significantly improved the production of indirect restorations, in particular CAD/CAM restorations. These types of restorations can be produced by milling of composite or ceramic blocks [1]. CAD/CAM composites offer some advantages, as they are more flexible and less fragile than glass ceramics [2]. CAD/CAM composites also have the advantage of being easily modifiable or repairable and they have appropriate stress-absorbing features.

CAD/CAM composites show lower abrasiveness than do ceramic materials, and they can be milled more quickly with less wear of milling tools [2]. Moreover, after milling they have been proven to result in crowns with smoother margins [3]. The reduced brittleness of the polymer-containing materials accounts for less chipping at the crown margins, which results in smoother margins [2, 3]. Many manufacturing companies have recently improved their CAD/CAM composite blocks by enhancing the filler technology and thus achieving better material properties [4].

The efficiency of CAD/CAM composite restoration can be evaluated by various laboratory-measuring parameters such as mechanical, bonding, and esthetic properties [5]. The shear-bond strength (SBS) test is the most commonly used test in measuring the material's bond strength, as it is the easiest and fastest method [6, 7]. Moreover, evidence indicates that mechanical testing of bonded interfaces has provided deeper knowledge of the materials' bonding performance and defined guidelines for their application [8, 9]. The strength and durability of the bond between indirect CAD/CAM restoration and the resin cement is of great clinical importance in terms of longevity of the treatment outcome [10, 11]. To enhance the bond strength, mechanical or chemical intervention that can increase the fitting surface irregularity of CAD/CAM restoration would be clinically useful [12]. Based on the restorative material, various surface treatment techniques have been suggested such as acid etching [5], air-particle abrasion [13] and grinding with a bur [14]. These techniques have resulted in improved bond strength of CAD/CAM restoration to resin cement by providing micro-mechanical retention [15].

The indirect restorative procedure has more bonding interfaces than the direct one; this fact can increase the difficulty of bonding the indirect restorations to the tooth structure [16]. Therefore, the luting agent or resin cement plays a key role in the longevity of the CAD/CAM treatment outcome [17]. Adequate polymerization of the resin cement is greatly important to guarantee satisfactory performance of the indirect restorations clinically [18]. Several parameters can affect the resin cement polymerization, including the composition of the resin cement, the shade, thickness, and translucency of the restoration [19]. Numerous experimental studies and theoretical calculations based on the Beer-Lambert law have reported that light transmission through restoration decreases as the thickness of the restoration increases [20, 21, 22], and that other factors such as the matrix composition and the filler type may influence translucency [20, 23]. Since the translucency has been identified as an important factor influencing the polymerization efficiency of the underlying resin cement, as well as influencing the esthetic outcome of the restoration [24], it is a crucial property to investigate.

In previous studies, experimental short fiber-reinforced (SFRC) CAD/CAM composites have shown promising mechanical, load-bearing, and surface characteristics [25, 26]. However, no information is available regarding the bonding and the translucency of this composite. Therefore, the current study aimed to evaluate the SBS of resin cement to SFRC CAD/CAM composite in comparison with different conventional CAD/CAM (Cerasmart 270), 3D printed (GC TEMP PRINT, Pro3dure GR-17), and laboratory (Gradia Plus) light-cured composites after different surface treatments. Furthermore, translucency parameter values and light transmission of composites at different thicknesses were evaluated.

MATERIAL AND METHODS

Materials preparation and light-curing protocol

The composition of the composites used in this study are detailed in Table 1. Experimental SFRC CAD/CAM composite blocks were prepared from a mixture of UDMA, TEGDMA (70/30) resins (23 wt%) with short (200-300 & Ø7 μm) glass fiber (25 wt%), and barium particulate glass (52 wt%). A high-speed mixing machine was used in mixing for 5 minutes (SpeedMixer DAC 400.1, 3500 rpm; Synergy Devices). The temperature during mixing was monitored by an infrared thermometer. SFRC CAD/CAM composite blocks were polymerized using an oven at 120°C for 60 minutes. For Cerasmart 270 (which was received fully polymerized) and SFRC CAD/CAM blocks, specimens were prepared by sectioning the blocks under water cooling using a low-speed diamond saw (Secotom-50; Struers). The 3D-printed specimens were made of Pro3dure GR-17 and GC TEMP PRINT composites (Table 1) by using a digital light processing printer (Asiga Max UV; Asiga). The printing was done according to the manufacturer's instructions. For the conventional laboratory composite (Gradia Plus), initial curing of specimens was performed using a hand light-curing device (Elipar TM S10; 3 M ESPE) for 20 seconds from different overlapping sections. The wavelength of the curing light was between 430 and 480 nm and its' intensity 1600 mW/cm^2. The light source was placed close with the composite surface (1-2 mm). After that, a light-curing oven (Targis Power; Ivoclar Vivadent) was used for further polymerization of the specimens for 3 minutes.

SBS test

For each of the five composites, 16 discs (14 mm length × 12 mm width × 3 mm thickness) were prepared and positioned in acrylic resin blocks (Palapress Vario; Heraeus Kulzer,). The surfaces were ground with 180 and 320 grit silicon carbide papers using an automatic grinding machine (Rotopol-1; Struers). The groups were then subdivided based on the surface treatment method used into hydrofluoric acid and air-particle abrasion subgroups (n = 8/group). Hydrofluoric acid gel 4.5% (Ivoclar Vivadent) was applied to the discs for 60 seconds followed by washing, air-drying, and application of primer (G-Multi Primer; Table 1). In the air-particle abrasion groups, the surface was air abraded using aluminium oxide particles (Ø 50 μm, pressure 30 psi), the specimens were subsequently washed and air-dried before the G-Multi Primer was applied.

TABLE 1 Materials used in the study

Material (type)	Manufacturer	Composition
TEMP PRINT medium (3D printed)	GC	UDMA, dimethacrylate, inorganic silica fillers <25 wt%
GR-17 temporary (3D printed)	Pro3dure Medical	Bismethacrylate and dimethacrylate monomers, silicon dioxide <50 wt%
Gradia Plus (conventional laboratory)	GC	UDMA, dimethacrylate, inorganic fillers (71 wt%), Prepolymerized fillers (6 wt%)
Cerasmart 270 (CAD/CAM)	GC	Bis-MEPP, UDMA, DMA, Silica (20 nm), barium glass (300 nm) 71 wt%
SFRC block (CAD/CAM)	Experimental	UDMA, TEGDMA, Short glass fiber (200-300 μm & Ø7 μm), Barium glass 77 wt%
G-Cem LinkForceXXX (Dual-cure resin cement)	GC	Bis-GMA, Bis-MEPP, UDMA, dimethacrylate, barium glass (300 nm), initiator, pigments
G-multi primer	GC	Ethyl alcohol (90-100%), phosphoric acid ester monomer (1-5%), dimethacrylate (1-5%)

Abbreviations: Bis-GMA, bisphenol-A-glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-MEPP, Bis (p-methacryloxy (ethoxy) 1–2 phenyl)-propane; wt%, weight percentage; vol%, volume percentage.

A custom-made transparent mold with a flat end (diameter 3.6 mm and height 3 mm) was positioned centrally on the flat composite surface. The luting cement (G-CEM linkForce; GC - Table 1) was applied through the mixing tip into the mold. After removing excess cement, the specimens were light-cured through the mold on each side and from the top for 20 seconds using the Elipar TM S10 (3 M ESPE device). Then, the mold was carefully removed and specimens were stored for 72 hours in water (37˚C) before testing.

The SBS test was used to identify the quality of adhesion between the luting cement and the composite materials. Firstly, the specimens were mounted in a mounting jig (Bencor Multi-T shear assembly; Danville Engineering). Thereafter, a shearing rod was placed parallel to and against the interface between the composite and the resin luting cement. Then, at room temperature (23 ± 1˚C) and a crosshead speed 1.0 mm/min a universal testing machine (Model LRX; Lloyd Instruments) was used to load all specimens until failure. Data were recorded by PC software (NEXYGEN; Lloyd Instruments). The bond strength was calculated by dividing the maximum load at failure (N) by the bonding area (mm^2). The results were recorded in megapascal (MPa).

Translucency measurement (TP)

A total of 75 specimens (14 length, 12 mm width) were prepared from the tested composites (n = 15/material). Specimens were subdivided according to thickness into three different 1, 2, 3 mm subgroups (n = 5/group). Curing of the composite was performed as mentioned previously. CIELAB color scale was used to determine the specimen color relative to the standard illuminant D65. Color assessment was done on a reflection spectrophotometer (CM-700d; Konica-Minolta) over a black tile (CIE $L^* = 0$, $a^* = 0.01$ and $b^* = 0.03$) and a white one (CIE $L^* = 99.25$, $a^* = -0.09$ and $b^* = 0.05$). Aperture diameter was 3 mm, and the viewing and illuminating configuration was CIE diffuse/10 geometry with specular component included (SCI) geometry. To obtain the translucency of the tested composite specimens, the color difference between the specimen on the white background and the specimen on the black one was measured using the following equation:

$$TP = [(L_W * - L_B *)^2 + (a_W * - a_B *)^2 + (b_W * - b_B *)^2]^{1/2}$$

where TP is the translucency parameter, the variable "W" refers to color coordinates on the white background and the variable "B" refers to color coordinates on the black background.

Light transmission analysis

For each tested composite, five discs at each of the three different composites thicknesses (1, 2, and 3 mm) were prepared (n = 15/material). Each specimen was flat polished using an automatic grinding machine (Rotopol-1; Struers) and silicon carbide papers from #1200- to #4000- grit at 300 rpm underwater cooling. A digital caliper (Mitutoyo) measured the final thickness within a range of ± 0.1 mm. Then, the specimens were cleaned ultrasonically (Quantrex 90; L&R Ultrasonic) in deionized water for 10 minutes and dried for 20 seconds before further evaluation. The disc specimens were then positioned over a spectrometer (MARC Resin Calibrator, Blue Light Analytics) and light-cured for 20 seconds by direct application of the curing unit (Elipar TM S10, 3 M ESPE) perpendicularly and centrally over the specimen's surface by means of a mechanical arm. During the specimen curing, a spectrometer measured the irradiance transmitted to the bottom of the specimens in real-time. As a control, a ring mold without composite resin at different thicknesses was used. The MARC system contains a NIST-reference miniature spectrometer (USB4000; Ocean Optics) with a 3648-element linear CCD array detector (TCD1304AP; Toshiba). A CC3 cosine corrector is a sensor with 4 mm diameter, designed to collect light radiation at around 180°, excluding optical interface problems, which are related to light collection sampling geometry. The irradiance at wavelength of 360-540 nm was considered for data recording.

Microscopic analysis

Failure modes of shear-bond test specimens were visually examined and analyzed using a stereomicroscope at magnification force 40 (Wild M3Z; Wild Heerbrugg). The failure modes were then classified either as adhesive failure between composite material and resin luting cement or cohesive type of failure within composite material.

The effect of surface treatment on CAD/CAM composites was evaluated by scanning electron microscopy (SEM) (JSM 5500; Jeol). Before observation, specimens were coated with a gold layer in a vacuum evaporator using a sputter coater (BAL-TEC SCD 050 Sputter Coater; Balzers).

Statistical analysis

Using SPSS version 23 (SPSS, IBM) the shear bond strength data and the translucency parameter values were summarized by their mean values (SD) and tested using two-way ANOVA followed by Tukey HSD test ($\alpha = .05$) to determine the significance of the type of composite used and the surface treatment protocol (bond strength), respectively, the specimen thickness (translucency). The actual effects of surface treatment protocol and composite type on SBS values were estimated using a dummy variable linear regression analysis, using the hydrofluoric acid treatment and the conventional CAD/CAM composite (Cerasmart 270) as reference groups. A robust estimate approach was used to assess the regression model, and the modified R^2 was used to assess model fit.

RESULTS

The SBS values of the tested composites after air-particle abrasion and hydrofluoric acid surface treatment are shown in Figure 1. Irrespective of surface treatment, the conventional laboratory composite (Gradia Plus) exhibited the highest bond strength value, and this amounted to 22.4 MPa when treated with air-particle abrasion, while conventional CAD/CAM composite (Cerasmart 270) showed the lowest value (8.2 MPa) when treated with hydrofluoric acid (Figure 1). Regarding the two CAD/CAM composites, SFRC showed higher SBS than Cerasmart 270 when treated with air-particle abrasion (13.4 MPa) and hydrofluoric acid (9.6 MPa), however, the difference was not statistically significant ($P > 0.05$).

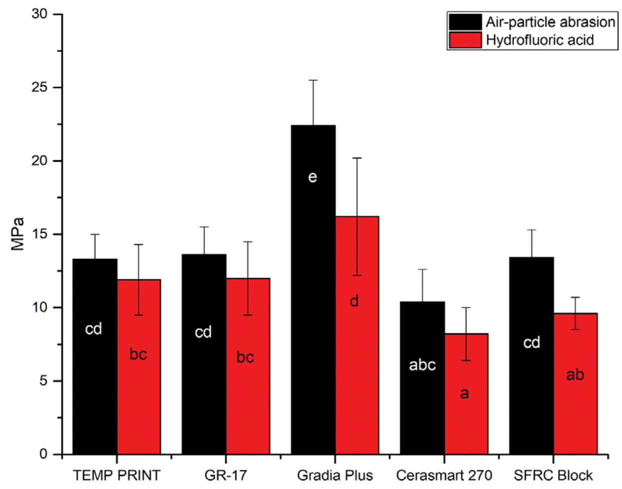

FIGURE 1 Shear-bond strength values (MPa) and standard deviations of the tested composites after different surface treatments. Groups denoted with the same letters are not statistically different ($P < 0.05$)

The results of the linear regression modeling of the SBS values as function of composite type and surface treatment protocol are presented in Table 2. Data indicated that both composite type and surface treatment protocol had a significant ($P < 0.05$; $R^2 = 0.65$) effect on SBS. The estimated bond strength value for conventional CAD/CAM composite (Cerasmart 270) treated with hydrofluroric acid was 8.49 MPa. Regardless of the composite used, air-particle abrasion resulted in SBS values that were on average 2.89 MPa higher ($P < 0.001$) than seen for hydrofluoric acid surface treatment (Table 2). The conventional laboratory composite (Gradia Plus) had a SBS value that was 9.38 MPa larger ($P < 0.001$) than the conventional CAD/CAM composite (Cerasmart 270), while the other types of composites had bond strength values that were 1.60 MPa (SFRC Block) to 2.70 MPa (TEMP PRINT) higher than seen for the conventional CAD/CAM composite (Cerasmart 270) (Table 2).

Table 3 presents the type of failures observed. When the failure mode of each composite was assessed, it was found that cohesive failure was predominant in 3D printed (TEMP PRINT and GR-17), and conventional CAD/CAM (Cerasmart 270) composite specimens, while with SFRC specimens the failure mode was 100% adhesive. As seen in Table 3 for laboratory composite (Gradia Plus), nearly half of the specimens exhibited cohesive failure, and the other half showed adhesive type of failure.

The translucency parameter values of the tested composites at different thicknesses are shown in Figure 2. Despite

TABLE 2 Results of multivariable linear regression analysis of shear-bond strength values as a function of composite type and surface treatment protocol

Predictor	Value	B	95% CI	Significance[a]
Composite type	Cerasmart 2170	Ref.	–	
	TEMP PRINT	2.70	(1.05, 4.36)	0.002
	GR-17	2.61	(0.87, 4.35)	0.004
	Gradia Plus	9.38	(7.73, 11.04)	<0.001
	SFRC Block	1.60	(−0.05, 3.26)	0.057
Surface treatment	Hydrofluoric acid	Ref.	–	–
	Air-particle abrasion	2.89	(1.80, 3.97)	<0.001
Constant		8.49	(7.27, 9.72)	<0.001

Abbreviations: Ref., Reference category; CI, Confidence Interval; B, Beta coefficient.
$R^2 = 0.65$.
[a]Test of H_0: B = 0.

TABLE 3 The distribution of bond failure mode according to material type and surface treatment protocol

	Adhesive		Cohesive	
Material (type)	**AA**	**HF**	**AA**	**HF**
TEMP PRINT (3D printed)	0	0	10	10
GR-17 (3D printed)	0	0	10	10
Gradia Plus (conventional laboratory composite)	0	9	10	1
Cerasmart 270 (CAD/CAM)	1	3	9	7
SFRC Block (CAD/CAM	10	10	0	0

Abbreviations: AA, air-particle abrasion; HF, hydrofluoric acid treatment.

the manufacturing and composition differences between the tested composites, the translucency parameter values significantly correlated with the thickness of composite. As thickness increased, the translucency parameter value decreased for each composite. Comparing between the composites used at 1-, 2-, and 3-mm thickness, conventional laboratory and CAD/CAM composites showed the highest translucency parameter values, while 3D printed and SFRC CAD/CAM composites showed similar values ($P < 0.05$).

Figure 3 demonstrates light transmission through different thicknesses of each composite and the control. The irradiance

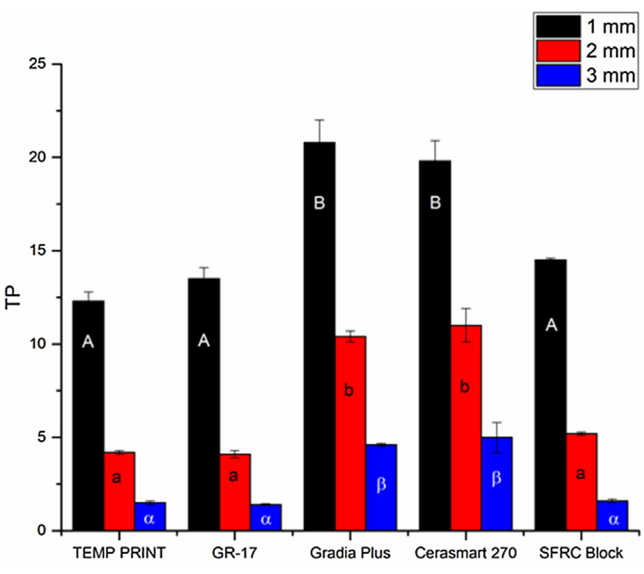

FIGURE 2 Translucency parameter mean values (TP) and standard deviations of the tested composites. Groups denoted with the same letters are not statistically different ($P < 0.05$)

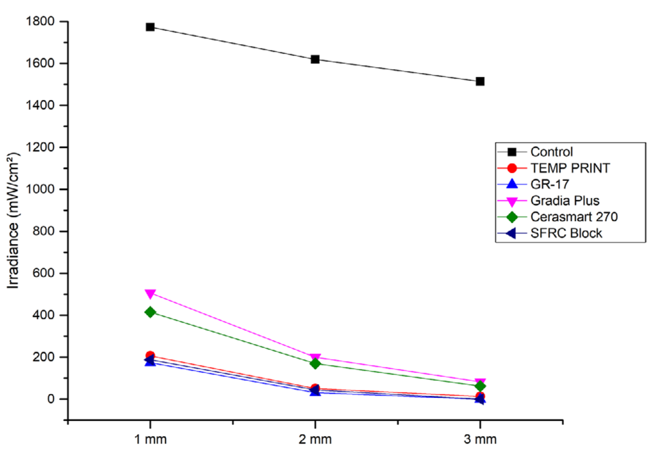

FIGURE 3 The light irradiance (mW/cm^2) of the transmitted light of curing unit at different thicknesses to the sensor through tested composites. Light curing without composite at different thicknesses was used as a control

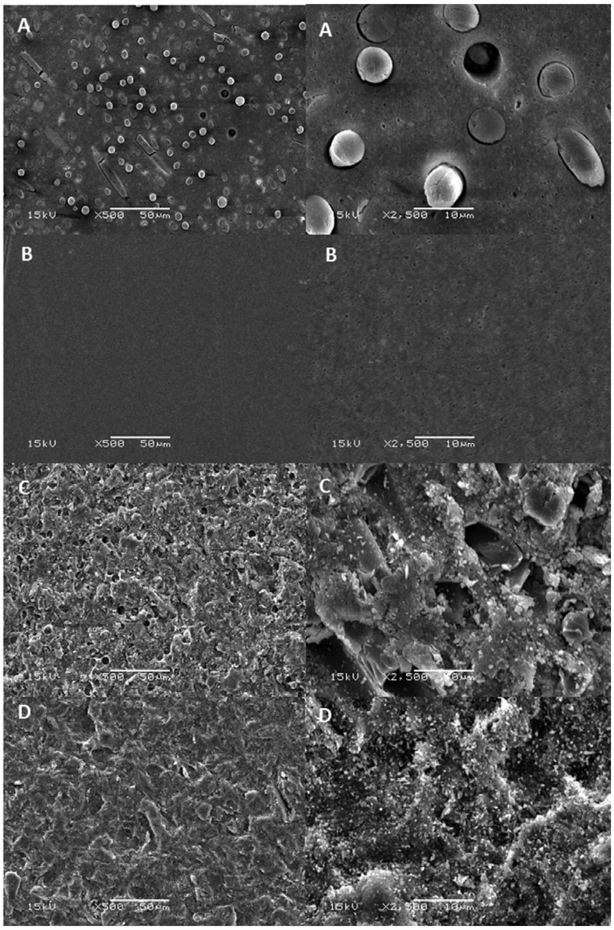

FIGURE 4 SEM images of CAD/CAM composites surfaces observed at different magnifications (500 & 2500x) after acid etching and air-particle abrasion. (A) Acid etched SFRC; (B) acid etched Cerasmart 270; (C) air-particle abraded SFRC; (D) air-particle abraded Cerasmart 270

values of penetrating light are in line with the translucency parameter values, as thickness increases, light transmission decreases for all composites. Light transmission through 3 mm thickness in all tested composites was either extremely low or did not exist at all. Conventional laboratory (Gradia Plus) and CAD/CAM (Cerasmart 270) composites again showed the highest values, while the other composites exhibited lower values. However, as in the translucency parameter results, the light irradiance of 3D printed and SFRC CAD/CAM composites were nearly equal at all three thicknesses.

Figure 4 shows SEM images for CAD/CAM composites after air-particle abrasion and hydrofluoric acid etching treatment under different magnifications. SEM images showed clearly the difference in appearance between surfaces treated with the two different treatment methods. Air-particle abrasion resulted in rougher surface than hydrofluoric acid in both CAD/CAM composite specimens. In SFRC composite treated with air-particle abrasion specimens, fibers are being pulled out and resulted in a pitted surface. While in hydrofluoric acid treated groups, fibers were partially dissolved from SFRC composite, leaving rough surface, and small porosities were seen at the conventional CAD/CAM composite surface.

DISCUSSION

The results of the present study have demonstrated that the tested composites made using various manufacturing methods and compositions differed in terms of the tested properties. The results of a SBS test indicated that both composite type and surface treatment influence the bonding strength to resin cement. To achieve better bonding performance, alterations need to be created in the material's surface texture; moreover, the selection of the resin cement type is of great importance [17]. Air-particle abrasion using aluminum oxide particles causes nonselective degradation of the composite, which creates irregular surface, while the acid etching technique using hydrofluoric acid works on dissolving the filler particles, which results in porous surface [27]. In this study, in addition to the surface treatment, G-Multi Primer was applied to the composite surface to enhance the bond strength of the composite to the resin luting cement. As shown in previous studies, the role of the primers is to increase the penetration of the luting cement monomers into composite, which can enhance the resulting adhesive interface [13, 28, 29].

Based on our results, air-particle abrasion resulted in higher SBS values than acid etching treatment for all tested composites. This could be explained by the fact that air-particle abrasion resulted in higher surface roughness than hydrofluoric acid (Figure 4), which increases the bonded surface area and surface energy; it also provides higher micro-mechanical retention at interface between resin cement and composite [30]. These findings are in line with previous studies [31–33], which proved that air-particle abrasion seems to be the best surface treatment option for indirect composites. On the other hand, some studies suggested that, hydrofluoric acid etching is the best surface treatment method for certain types of CAM/CAM composites [34, 35]. Other investigations have proved that air-particle abrasion could cause damage to the surface and cracks to the subsurface, which compromises restoration long-term durability; moreover, the exposure of restoration margins to air-particle abrasion could result in marginal gap, which is unacceptable clinically [36].

In the present study, as a general tendency, the conventional laboratory composite (Gradia Plus) exhibited the highest SBS values when treated with either air-particle abrasion or hydrofluoric acid. Conventional laboratory composite is not as highly polymerized as CAD/CAM manufactured composites, thus there is a high percentage of residual or unreacted monomers. These monomers can interact chemically with monomers usually present in the resin cement. Theoretically, it is possible that methacrylate monomers in resin luting cement can form a chemical bond with the composite material, and this bond is enhanced by applying the primer. Methacrylate monomers of the resin luting cement may copolymerize with unreacted C = C double bonds of the composite material when exist [37]. Another possible explanation for high SBS values with the conventional laboratory composite could be due to the high fillers content (77 wt%) in particular the large sized pre-polymerized fillers. Both surface treatment techniques have a significant influence on the large size fillers [38], creating a highly irregular surface and this might in turn result in a higher bond strength [30]. This is in agreement with MIYAZAKI et al. [39], who found in their in vitro research that bond strength increases with increasing filler content. This could explain the low SBS values of 3D printed composites (TEMP PRINT and GR-17). Although they contain residual monomers, they have a lower content of small sized particulate fillers (<50 wt%), which reduce the effect of surface treatment. In addition, the low filler content causes weakness of the composite matrix; thus, shearing stresses cannot be withstood. Corroborating this interpretation, we observed that 100% of failures among the 3D printed composites were of the cohesive type (Table 3).

Although CAD/CAM manufactured composites (SFRC and Cerasmart 270) have high filler content, they exhibited SBS values lower than those of the conventional laboratory composite. CAD/CAM composites are polymerized under high temperature and high pressure with photo-curing and heat-curing. Polymerization under optimum conditions results in a higher degree of conversion and less residual monomers. Though this can improve the mechanical properties of the composite [25, 40] it results in lower chemical bond or copolymerization between the CAD/CAM composite and resin cement and subsequently lower SBS. It has been stated in literature that the bonding of a new composite resin to a highly polymerized thermoset matrix is weak because of the cross-linked nature of the thermoset matrix [41, 42]. If, however, the matrix is of the semi-IPN (interpenetrating polymer network) type, the bonding possibilities during cementation or repair improve, and this is due to monomer diffusion or interpenetration, which is possible only for the semi-IPNs [43].

The fiber diameter in the SFRC CAD/CAM composite is larger than the diameter of the Cerasmart 270 fillers; this may explain the rougher surface seen after air abrasion as shown in the SEM images (Figure 4C, D), and in turn it explains the higher SBS values. In hydrofluoric acid-treated surfaces (Figure 4A, B), we observed the effect of etching depends on the filler composition and size [38]. The small surface porosities observed in Cerasmart 270 specimens treated with hydrofluoric acid were probably resulting from dissolved fillers in the matrix. While with the SFRC composite, the glass fibers were partially dissolved, leaving rough and higher surface area for bonding. This observation corroborates reports that 4.5 % hydrofluoric acid treatment can have considerable effects on glass fibers [44].

Differences were noted regarding the failure mode of SFRC and Cerasmart 270 composites (Table 3). These variations could be explained as resulting from differences in the filler content and the polymer matrices of these CAD/CAM composites. Short fiber fillers can provide high toughness to the matrix to resist the shearing stress [45] and consequently it was the only tested composite showing no cohesive failure at all. On the other hand, Cerasmart 270 is a brittle material with lower toughness than the SFRC composite, therefore, the majority of failures were of the cohesive type [25]. Furthermore, its matrix could be weakened after surface treatment, and this can lead to cohesive type of failure as well.

Our results clearly demonstrated that the translucency parameter values and light transmission were influenced by the thickness of each tested composite. A material's translucency and the light transmitted through different composites depends on the scattering and absorption coefficient [46]. Interestingly, although they have low filler content, the 3D printed composites had lower translucency parameter and light transmission values (Figures 2 and 3). The matrix of the 3D printed composites contains residual monomers, which may lead to decrease in the homogeneity of the inner structure. This structure possibly creates internal interfaces, on which light would be scattered, broken, or reflected, and in turn lower translucency parameter values and light transmission [47]. While the conventional laboratory composite showed higher translucency parameter and transmission values, this could be due to good matching in refractive index between the matrix and the filler particles [48].

Although the glass fibers in SFRC CAD/CAM composite can conduct light [49], the translucency parameter and light transmission values were lower than seen for Cerasmart 270. This could be attributed to the small nanoparticle fillers in Cerasmart 270, while the SFRC composite has larger fillers including microscale fibers. According to the Rayleigh equation, the particles size has a great influence, predicting that larger size particles would cause a decrease in transmission [50]. The interaction of light with the smaller particles in Cerasmart 270 results in less light absorbance and consequently higher translucency parameter values [51]. In addition, differences in the amount of filler in each composite could cause this variation. With less matrix and high filler content (Table 1), translucency decreases due to light scattering [52]. Although the SFRC composite showed acceptable translucency parameter values, it is still an experimental material, and further optimization is needed to improve its translucency.

The findings of this study have to be seen in light of some limitations. The SBS test was used to determine the bond strength of the materials, where the tensile-bond strength could be more accurate in detecting bond strength differences between materials [53]. Moreover, the SBS was evaluated after a short-term water storage (72 hours) and to understand the bond durability of composites to resin cement, long-term water storage would be required. To gain more knowledge about the experimental SFRC CAD/CAM composite, further research is needed to clarify the effect of aging (thermal, mechanical, and chemical) on the surface integrity and bonding performance of this composite.

Within the limitations of the present study, the following conclusion can be drawn:

(i) Conventional laboratory composite exhibited superior SBS and optical properties compared to CAD/CAM and 3D printed composites.
(ii) The SBS of short fiber-reinforced CAD/CAM composite to resin cement was higher than that of conventional CAD/CAM composite.
(iii) Air-particle abrasion of all tested composites was more effective in enhancing SBS than was hydrofluoric acid etching.

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CONFLICTS OF INTEREST

All authors agreed with the concept of the manuscript and they confirm that there is no economic benefit or any financial interest to report. Pekka Vallittu declares that he is a consultant for Stick Tech—Member of GC in training and research and development. Other authors do not have conflicts of interests.

AUTHOR CONTRIBUTIONS

Conceptualization: Sufyan Garoushi, Lippo Lassila; **Investigation**: Enas Mangoush; **Formal analysis**: Enas Mangoush, Lippo Lassila; **Methodology**: Lippo Lassila; **Data curation**: Lippo Lassila; **Writing-original draft**: Enas Mangoush; **Writing- review & editing**: Pekka K. Vallittu, Sufyan Garoushi; **Supervision**: Pekka K. Vallittu, Sufyan Garoushi.

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REFERENCES

1. Suzaki N, Yamaguchi S, Hirose N, Tanaka R, Takahashi Y, Imazato S, Hayashi M. Evaluation of physical properties of fiber-reinforced composite resin. Dent Mater. 2020;36:987–996.
2. Awada A, Nathanson D. Mechanical properties of resin-ceramic CAD/CAM restorative materials. J Prosthet Dent. 2015;114:587–593.
3. Tsitrou EA, Northeast SE, van Noort R. Brittleness index of machinable dental materials and its relation to the marginal chipping factor. J Dent. 2007;35:897–902.
4. Sripetchdanond J, Leevailoj C. Wear of human enamel opposing monolithic zirconia, glass ceramic and composite resin: an in vitro study. J Prosthet Dent. 2014;112:1141–1150.
5. Stawarczyk B, Liebermann A, Eichberger M, Güth GF. Evaluation of mechanical and optical behavior of current esthetic dental restorative CAD/CAM composites. J Mech Behav Biomed Mater. 2015;55:1–11.
6. Burke FJT, Hussain A, Nolan L, Fleming GJP. Methods used in dentine bonding tests: an analysis of 102 investigations on bond strength. Eur J Prosthodont Rest Dent. 2008;16:158–165.
7. Sudsangiam S, van Noort R. Do dentin bond strength test serve a useful purpose? J Adhes Dent. 1999;1:57–67.
8. Shono Y, Ogawa T, Terashita M, Carvalho RM, Pashley EL, Pashley DH. Regional measurement of resin-dentin bonding as an array. J Dent Res. 1999;78:699–705.
9. Hashimoto M, Tay FR, Svizero NR, de Gee AJ, Feilzer AJ, Sano H, et al. The effects of common errors on sealing ability of total-etch adhesives. Dent Mater. 2006;22:560–568.
10. Cekic-Nagas I, Ergun G, Egilmez F, Vallittu PK, Lassila LVJ. Micro-shear bond strength of different resin cements to ceramic/glass-polymer CAD-CAM block materials. J Prosthodont Res. 2016;60:265–273.
11. Lise DP, Van Ende A, De Munck J, Vieira L, Baratieri LN, Van Meerbeek B. Microtensile bond strength of composite cement to novel CAD/CAM materials as a function of surface treatment and aging. Oper Dent. 2017;42:73–81.
12. Piascik JR, Wolter SD, Stoner BR. Development of a novel surface modification for improved bonding to zirconia. Dent Mater. 2011;27:99–105.
13. Higashi M, Matsumoto M, Kawaguchi A, Miura J, Minamino T, Kabetani T, et al. Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin blocks. Part 1: effects of sandblasting and silanization. Dent Mater J. 2016;35:21-28.
14. Gungor MB, Nemli SK, Bal BT, Ünver S, Doğan A. Effect of surface treatments on shear bond strength of resin composite bonded to CAD/CAM resin-ceramic hybrid materials. J Adv Prosthodont. 2016;8:259–266.
15. Spitznagel FA, Horvath SD, Guess PC, Blatz MB. Resin bond to indirect composite and new ceramic/polymer materials: a review of the literature. J Esthet Restor Dent. 2014;26:382–393.
16. Elsaka SE. Bond strength of novel CAD /CAM Restorative materials to self-adhesive resin cement: the effect of surface treatments. J Adhes Dent. 2014;16:531–540.
17. Stawarczyk B, Awad D, Ilie N. Blue-light transmittance of esthetic monolithic CAD/CAM materials with respect to their composition, thickness, and curing conditions. Oper Dent. 2016;41:531–540.
18. Meng X, Yoshida K, Atsuta M. Influence of ceramic thickness on mechanical properties and polymer structure of dual-cured resin luting agents. Dent Mater. 2008;24:594–599.

19. Sulaiman TA, Abdulmajeed AA, Donovan TE, Ritter AV, Lassila LV, Valittu PK, Närhi TO. Degree of conversion of dual-polymerizing cements light polymerized through monolithic zirconia of different thicknesses and types. J Prosthet Dent. 2015;114:103–108.
20. Garoushi S, Vallittu P, Shinya A, Lassila L. Influence of increment thick- ness on light transmission, degree of conversion and micro hardness of fill bulk|composites. Odontology. 2016;104:291–297.
21. Meng X, Yoshida K, Atsuta M. Hardness development of dual-cured resin cements through different thicknesses of ceramics. Dent Mater J. 2006;25:132–137.
22. Lassila LV, Nagas E, Vallittu PK, Garoushi S. Translucency of flowable bulk-filling composites of various thicknesses. Chin J Dent Res. 2012;15:31–35.
23. Azzopardi N, Moharamzadeh K, Wood DJ, Martin N, van Noort R. Effect of resin matrix composition on the translucency of experimental dental composite resins. Dent Mater. 2009;25:1564–1568.
24. Ilie N. Altering of optical and mechanical properties in high-translucent CAD-CAM resin composites during aging. J Dent. 2019;85:64–72.
25. Mangoush E, Garoushi S, Vallittu PK, Lassila L. Influence of short fiber- reinforced composites on fracture resistance of single-structure restorations. Eur J Prosthodont Restor Dent. 2020;28:189–198.
26. Mangoush E, Lassila L, Vallittu PK, Garoushi S. Microstructure and surface characteristics of short- fiber reinforced CAD/CAM composite blocks. Eur J Prosthodont Restor Dent. 2020;28:1–9.
27. Park JH, Choi YS. Microtensile bond strength and micromorphologic analysis of surface-treated resin nanoceramics. J Adv Prosthodont. 2016;8:275–284.
28. Khan AA, Al-Kheraif AA, Mohamed BA, Perea-Lowery L, Säilynoja E, Vallittu P. Influence of primers on the properties of the adhesive interface between resin composite luting cement and fiber-reinforced composite. J Mech Behav Biomed Mater. 2018;88:281–287.
29. Khan AA, Mohamed BA, Al-Shamrani SS, Ramakrishnaiah R, Perea-Lowerye L, Säilynoja E, Vallittu P. Influence of monomer systems on the bond strength between resin composites and polymerized fiber-reinforced composite upon Aging. J Adhes Dent. 2019;21:509–516.
30. Fonseca R, Haneda I, Almeida-Júnior A, Abi-Rached F, Adabo G. Efficacy of air-abrasion technique and additional surface treatment at titanium/resin cement interface. J Adhes Dent. 2012;14:453–459.
31. Arpa C, Ceballos L, Fuentes MV, Perdigão J. Repair bond strength and nano-leakage of artificially aged CAD-CAM composite resin. J Prosthet Dent. 2019;121:523–530.
32. Ali A, Takagaki T, Nikaido T, Abdou A, Tagami J. Influence of ambient air and different surface treatments on the bonding performance of a CAD/CAM composite block. J Adhes Dent. 2018;20:317–324.
33. Soares CJ, Soares PV, Pereira JC, Fonseca RB. Surface treatment protocols in the cementation process of ceramic and laboratory-processed composite restorations: a literature review. J Esthet Restor Dent. 2005;17:224–235.
34. Abdou A, Takagaki T, Alghamdi A, Tichy A, Nikaido T, Tagami J. Bonding performance of dispersed filler resin composite CAD/CAM blocks with different surface treatment protocols. Dent Mater J. 2021;40:209–219.
35. Yanardag E, Yilmaz S, Karakaya E, Ongun S. Effect of different surface treatment methods on micro-shear bond strength of CAD-CAM restorative materials to resin cement. J Adhes Sci Tech. 2018;33:1–14.
36. Yoshihara K, Nagaoka N, Maruo Y, Nishigawa G, Irie M, Yoshida Y. Sandblasting may damage the surface of composite CAD-CAM blocks. Dent Mater. 2017;33:124–135.
37. Stawarczyk B, Basler T, Ender A, Roos M, Ozcan C. Effect of surface conditioning with airborne particle abrasion on the tensile strength of polymeric CAD/CAM crowns luted with self-adhesive and conventional resin cements. J Prosthet Dent. 2012;107:94–101.
38. Loomans BAC, Cardoso MV, Opdam NJM, Roeters FJM, De Munck J, Huysmans MCDNJM, Meerbeek BV. Surface roughness of etched composite resin in light of composite repair. J Dent. 2011;39:499–505.
39. Miyazaki M, Hinoura K, Onose H, Moore BK. Effect of filler content of light-cured composites on bond strength to bovine dentine. J Dent. 1991;19:301–303.
40. Garoushi S, Vallittu PK, Lassila LV. Fracture resistance of short, randomly oriented, glass fiber-reinforced composite premolar crowns. Acta Biomater. 2007;3:779–784.
41. Frese C, Decker C, Rebholz J, Stucke K, Staehle HJ, Wolff D. Original and repair bond strength of fiber-reinforced composites in vitro. Dent Mater. 2014;30:456-462.
42. Wolff D, Geiger S, Ding P, Staehle HJ, Frese C. Analysis of the interdiffusion of resin monomers into pre-polymerized fiber-reinforced composites. Dent Mater. 2012;28:541–547.
43. Vallittu PK. Interpenetrating polymer networks (IPNs) in dental polymers and composites. J Adhesion Sci Technol. 2009;23:961–972.
44. Aksornmuang J, Chuenarrom CH, Chittithaworn N. Effects of various etching protocols on the flexural properties and surface topography of fiber-reinforced composite dental posts. Dent Mater J. 2017;36:614–621.
45. Garoushi S, Hatem M, Lassila L, Vallittu PK. The effect of short fiber composite base on microleakage and loadbearing capacity of posterior restorations. Acta Biomater Odontol Scand. 2015;1:6–12.
46. Musanje L, Darvell BW. Curing-light attenuation in filler-resin restorative materials. Dent Mater. 2006;22:804–817.
47. Güth JF, Zuch T, Zwinge S, Engels J, Stimmelmayr M, Edelhoff D. Optical properties of manually and CAD/CAM-fabricated polymers. Dent Mater J. 2013;32:865–871.
48. Shortall AC, Palin WM, Burtscher P. Refractive index mismatch and monomer reactivity influence composite curing depth. J Dent Res. 2008;87:84–88.
49. Garoushi S, Vallittu P, Lassila L. Depth of cure and surface microhardness of experimental short fiber-reinforced composite. Acta Odontol Scand. 2008;66:38–42.
50. Masotti AS, Onofrio AB, Conceicao EN, Spohr AM. UV–Visspectrophotometric direct transmittance analysis ofcomposite resins. Dent Mater. 2007;23:724–730.
51. Lee YK. Influence of filler on the difference between the transmitted and reflected colors of experimental resin composites. Dent Mater. 2008;24:1243–1247.
52. Lise DP, Ende AV, De Munck J, Yoshihara K, Nagaoka N, Vieira L, Meerbeek BV. Light irradiance through novel CAD–CAM block

materials and degree of conversion of composite cements. Dent Mater. 2018;34:296–305.

53. El Zohairy AA, Saber MH, Abdalla AL, Feilzer AJ. Efficacy of microtensile versus microshear bond testing for evaluation of bond strength of dental adhesive system to enamel. Dent Mater. 2010;26:848–854.

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