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Microstructure and Surface Characteristics of Short-Fiber Reinforced CAD/CAM Composite Blocks

Keywords

Wear
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Surface Gloss

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ABSTRACT

Objective: The aim was to investigate certain surface properties and microstructure of an experimental short fiber-reinforced CAD/CAM composite block (SFRC) in comparison with different CAD/CAM, 3D-printing and manually-made commercial composites (Cerasmart 270, GC Temp PRINT, Pro3dure GR-17, Essentia U, Gradia Plus and everX Flow). Methods: A wear-test was performed using a chewing-simulator with 15000 cycles. Wear depth (n=6) was assessed by 3D optical-profilometer. Surface roughness (SR) before and after wearing-test was evaluated. A Vickers-indenter was utilized for evaluating surface microhardness (VH) and glossmeter was utilized to measure the surface gloss at 60°. The surface microstructure of each composite was investigated with SEM. Data were statistically analyzed with analysis of variance ANOVA (p=0.05). Results: Significant differences in the surface properties were found according to the type of composite (p<0.05). Cerasmart 270 exhibited the highest VH (94.8 V) and lowest SR (0.18 Ra) values (p<0.05) among the composites tested. The lowest wear depth measurement was located for GC Temp PRINT (19.3 μm) which was not significantly different (p>0.05) from Cerasmart 270 (20.7 μm). Conclusion: Incorporation of fibers to the composite of the CAD/CAM block did not negatively influence the surface characteristics of composite.

INTRODUCTION

Recent progress in the technology and research of new restorative and prosthetic materials has widened the choice for esthetic monolithic restorations. In view of progress in computer aided design / computer aided manufacturing (CAD/CAM) technologies, the production process of dental prosthesis using ceramic blocks for CAD/CAM processing is simple and reduced time to manufacture it contrary to the traditional heat-pressing technique of ceramics.[1] These blocks enable dental prostheses to be fabricated by milling directly after scanning the prepared teeth in patient's mouth. Thereby, the definitive restoration can be cemented inside the patient's mouth within short time if only a monolithic, i.e. single structure restoration is demanded. However, for the reasons that all-ceramics are brittle and prone to fatigue fracture in cyclic loading,[1-3] an attempt to find alternative to all-ceramic restorations was achieved with indirect resin composite restorations. Different from ceramics, composites are simple to construct, and promote less abrasion of the opposing dentition.[4,5] Many investigations have been focused on the clinical achievement of composite restorations made by either the manual build-up technique or CAD/CAM technique.[6-8] The major cause for failure in all studies was a catastrophic fracture, implying that the toughness of composite restorations is a critical property for achieving a good clinical result. The brittleness of composites is caused by a low resistance to crack extension and in most cases, these materials fail due to unstable crack propagation.[9]

Research has been conducted to find ways to improve the toughness and durability of final large composite restorations. Until this time and because of their improved fracture toughness and ability to arrest crack propagation, composites reinforced with short randomly oriented glass fibres (SFRCs) are interesting materials.[10-12] In view of the previous information and with the aim of looking for a novel SFRC CAD/CAM block that can substantially increase the toughness and strength of single-structure restorations, we developed and evaluated the fracture behavior related properties of the new SFRC CAD/CAM block in our previous research.[13] However, surface properties such as wear, roughness, microhardness and gloss of the SFRC CAD/CAM block is still not known. Therefore, the aim of this study was to investigate certain surface characteristics and microstructure of the new experimental SFRC CAD/CAM block in comparison with different CAD/CAM, 3D-printing and manually-made commercial dental composites (Cerasmart 270, GC Temp PRINT, Pro3dure GR-17, Essentia U, Gradia Plus and everX Flow).

The research hypothesis was that material type will have no effect on the surface related characteristics of composites.

MATERIALS AND METHODS

Table 1 contains the materials used in this study.

Experimental SFRC CAD/CAM composite blocks were prepared from mixture of UDMA, TEGDMA (70/30) resins (23 wt %) with short (200-300 µm & Ø7 µm) glass fiber (25 wt %) and barium particulate glass (52 wt %). The mixing was carried out by using a high speed mixing machine for 5 min (SpeedMixer DAC 400.1, 3500 rpm). Temperatures during the mixing were monitored by an infrared thermometer. Polymerization was performed using an oven at 120°C for 1 hour.

A total of 70 block-shaped specimens (14 mm length × 12 mm width × 2 mm thick) were prepared (n=10/material). Each specimen was polished flat using silicon carbide papers from #1200- to #4000-grit at 300 rpm and under water cooling using an automatic grinding machine (Rotopol-1; Struers). The final thickness (± 0.1 mm) was measured by a digital caliper (Mitutoyo Corp). Then, the specimens were cleaned ultrasonically (Quantrex 90, L&R Ultrasonics) in deionized water for 10 minutes and dried for 20 seconds before further evaluation.

Curing of the manually made composites (Essentia U, Gradia Plus and everX Flow) was done for 20 s by a hand light-curing device (Elipar TM S10, 3M ESPE) from one side of the mold in five different overlapping sections. The light wavelength was between 430 and 480 nm and light intensity was 1600 mW/cm^2. The light source was placed in close contact (1-2 mm)

Table 1. The composite materials used in the study

Material (type)	Manufacturer	Composition
TEMP PRINT medium (Printing)	GC	UDMA, dimethacrylate, inorganic silica fillers < 25 wt%
GR-17 temporary (Printing)	Pro3dure Medical,	Bismethacrylate and dimethacrylate monomers, silicon dioxide < 50wt%
Essentia Universal (Chair-side)	GC	UDMA, BisEMA, BisGMA, TEGDMA, Bis-MEPP, Prepolymerized silica and barium glass 81 wt%
Gradia Plus (Lab)	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%
everX Flow (Chair-side)	GC	Bis-EMA, TEGDMA, UDMA, Short glass fiber (200-300 µm & Ø7 µm), Barium glass 70 wt%
SFRC Block (CAD/CAM)	Experimental	UDMA, TEGDMA, Short glass fiber (200-300 µm & Ø7 µm), Barium glass 77 wt%

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

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with the composite surface. Specimens of Gradia Plus were further polymerised in a light-curing oven (Targis Power, Ivoclar Vivadent) for 3 min. Two groups of specimens were 3D-printed made of GC Temp PRINT and Pro3dure GR-17 composites by using a digital light processing printer (Asiga Max UV, Asiga) according to the manufacturers' instructions. For CAD/CAM materials (Cerasmart 270 and SFRC Blocks), the block-shaped specimens were prepared using a low-speed diamond saw (Struers).

TWO-BODY WEAR

Two specimens of each composite were placed in acrylic resin block to test the localized wear. After storing in water (37°C) for one day, 2-body wear test was accomplished using the chewing simulator (CS- 4.2, SD Mechatronik) that has two chambers simulating the horizontal and vertical movements concurrently with water. Each chamber composed of an upper sample holder that fasten the loading tip with a screw and a lower sample holder made of plastic where the composite specimen was embedded. The specimens were embedded in the lower sample holder in acrylic resin to be used as antagonistic wear material. The manufacturer's standard loading balls (Steatite ball, Ø 6 mm) were embedded in acrylic resin in the upper sample holders, and a fastening screw was used to fix them. A weight of 2 kg, which is comparable to 15.000 loading cycles with frequency of 1.5 Hz and a 20 N of chewing force were used. The wear patterns (n=6) on the surface of every specimen were profiled using a three-dimensional (3D) non-contact optical profilometer (Bruker Nano GmbH) with Vision64 software (Bruker Nano GmbH). The maximum wear depth values (µm), which represent the average of deepest points of all profile scans were measured from different points (*Figure 1*).

SURFACE ROUGHNESS

The surface roughness of each composite was measured before the wearing test and after it using a 3D non-contact optical profilometer (Bruker Nano GmbH). A 5× objective lens and a 0.5 multiplier was used, with back scan and length parameter of 20 µm and 60 µm in VSI/VXI mode to obtain a 3D rendering of the specimen surfaces (n=6). The Vision 64 software was used to generate roughness and surface areas parameters. Ra (roughness average) was considered to be the particular parameter of interest which measured from a mean line within the sampling length (*Figure 2*).

SURFACE MICROHARDNESS

The surface microhardness was measured for each composite by a Struers Duramin hardness microscope (Struers), with a load of 1.96 N applied for 10 s and a 40 objective lens. The specimen's surface of each composite (n=6) was subjected to 3 indentations. The diagonal length impressions were measured and Vickers values were changed to hardness values by the machine. The following equation was used to measure the surface hardness:

$$\mathrm{H} = \frac{1854.4 \times P}{d^2}$$

H is Vickers hardness in kg/mm^2, d is the length of the diagonals in µm and P is the load in grams.

SURFACE GLOSS

The surface gloss from the polished block-shaped specimens' of each composite (n=6) was determined at 60° incidence angle, by using a calibrated infrared Zehntner-Glossmeter (GmbH Testing Instruments) with an average of three measurements was recorded per specimen.

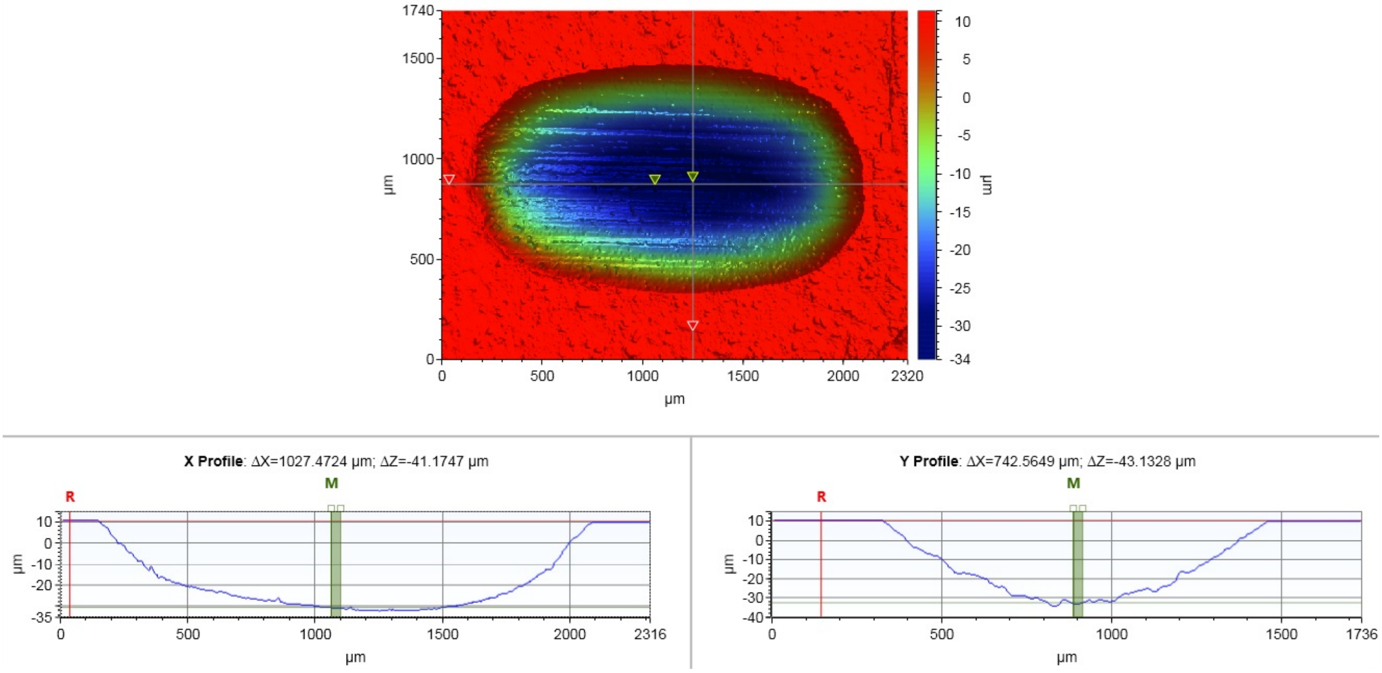

Figure 1: An example of 3D surface profile of the wear pattern.

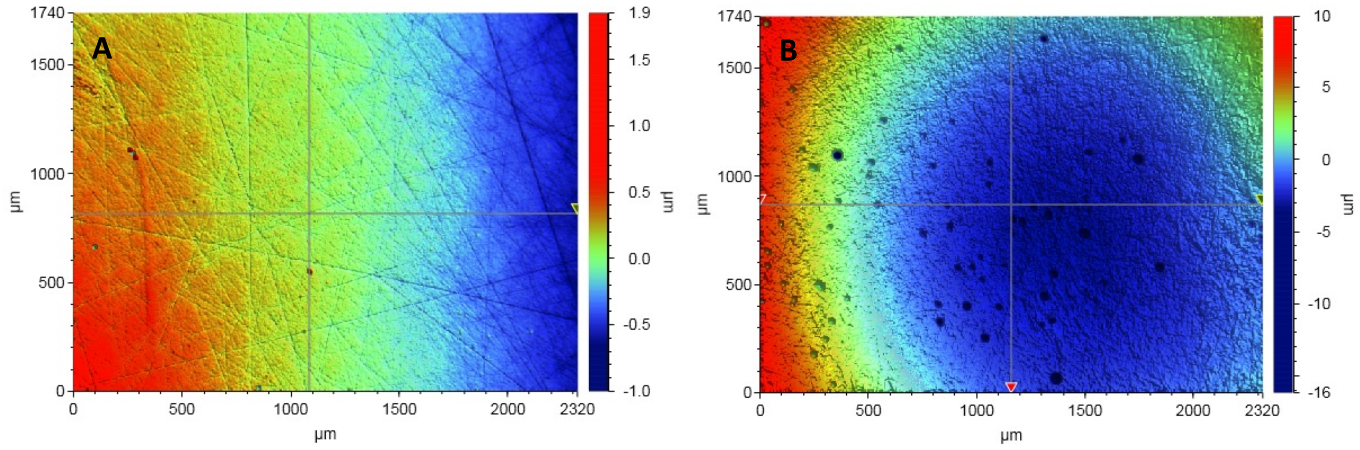

Figure 2: An example of 3D surface roughness profile of specimens before (A) and after wear test (B).

MICROSCOPIC ANALYSIS

Scanning electron microscopy (SEM, LEO) and energy-dispersive spectroscopy (EDS, LEO) have provided the characterisation of the microstructure and major elemental composition of the material's surface of the investigated materials (magnification: 5000 x). Polished specimens (n=2) from each composite were stored in a desiccator for one day. Then, they were coated with a gold layer using a sputter coater in a vacuum evaporator (BAL-TEC SCD 050 Sputter Coater) before the SEM/EDS examination. SEM/EDS observations were conducted at an operating voltage of 8 kV and working distance of 13 mm.

STATISTICAL ANALYSIS

The data were analyzed using SPSS version 23 (SPSS, IBM Corp.) using one-way (for surface microhardness, gloss and wear) and two-way (for surface roughness before and after wearing) analysis of variance (ANOVA) at the $p<0.05$ significance level followed by a Tukey HSD *post hoc* test to determine the differences between the groups.

RESULTS

The results of surface microhardness, roughness, gloss and wear were presented in Table 2 and Figure 3. Significant differences in surface properties were observed in accordance with the type of material ($p<0.05$), though, some interaction existed among the materials. As seen in Figure 3, the lowest wear depth measurement was located for GC Temp PRINT (19.3 µm) which was not significantly different ($p>0.05$) from Cerasmart 270 (20.7 µm). Gradia Plus presented the highest values (43 µm) of wear depth ($p<0.05$) compared with all tested composites *(Figure 3*). Cerasmart 270 exhibited the highest surface microhardness (94.8 V), while 3D-printing composites showed the lowest among the tested composites ($p<0.05$). Regardless of the material used, surface roughness (Ra) after wearing test was significantly higher (ranged between 4-11 µm) than before wearing test (ranged between 0.18-0.5 µm) ($p<0.05$). Cerasmart 270 presented the lowest Ra values before and after wearing test ($p<0.05$). The 3D images before and after wearing test are shown in Figure 2. Substantial quantity of small pits were seen as a result of filler particle exfoliation after wearing test *(Figure 2B*).

Table 2. Tested properties mean values and standard deviations (SD) of investigated composites

Materials	Surface microhardness (VH)	Surface roughness Ra (µm) before and after wearing		Gloss (GU)
TEMP PRINT	27.8 (1.0)^{a}	0.51 (0.05)^{e}	4.6 (0.5)^{b}	56.8 (1.3)^{b}
GR-17	31.1 (1.6)^{a}	0.25 (0.06)^{b}	5.4 (0.7)^{bc}	26.5 (1.2)^{a}
Essentia U	50.1 (2.0)^{b}	0.35 (0.06)^{c}	5.9 (1.6)^{c}	59.6 (1.2)^{b}
Gradia Plus	44.5 (0.7)^{b}	0.34 (0.04)^{c}	10.8 (0.3)^{f}	76.9 (2.4)^{c}
Cerasmart 270	94.8 (1.5)^{e}	0.18 (0.04)^{a}	4.2 (0.1)^{a}	83.4 (0.9)^{d}
everX Flow	59.2 (2.9)^{c}	0.46 (0.07)^{de}	8.4 (0.9)^{de}	75.8 (2.2)^{c}
SFRC Block	85.0 (2.9)^{d}	0.28 (0.08)^{bc}	7.5 (1.1)^{d}	81.0 (5.5)^{cd}

Same superscript letter above the values in a column indicates groups that were not statistically different ($p>0.05$).

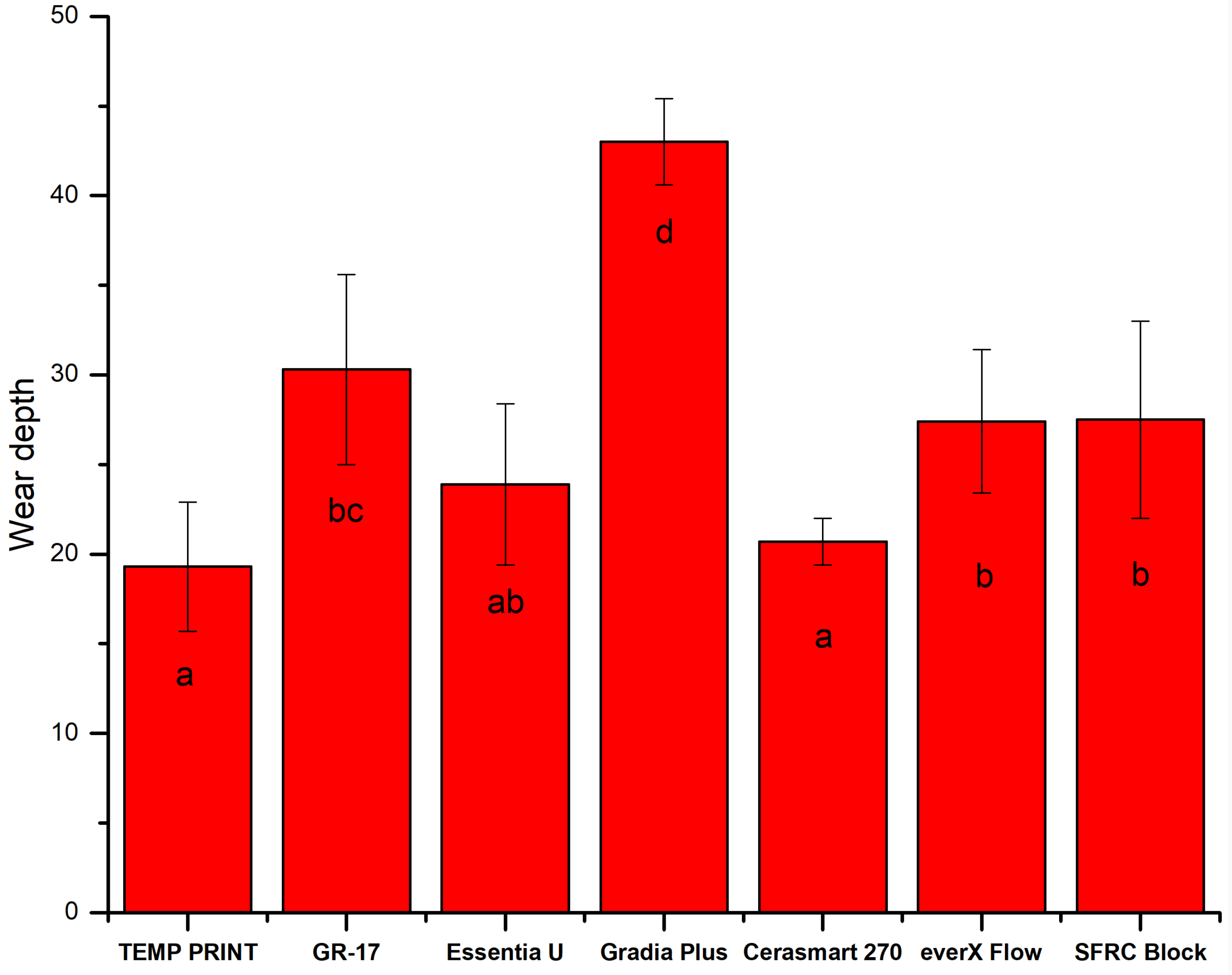

Figure 3: Bar graph illustrating means of wear depth (µm) and standard deviations (SD) of investigated composites. Different letters inside bars indicate significant differences ($p<0.05$).

Regarding the surface gloss, Cerasmart 270 showed the highest gloss value (83.4 GU) which was not significantly different ($p>0.05$) from Experimental SFRC block (81 GU).

Major elemental composition of each investigated composite determined with EDS is presented in Table 3. SEM/EDS examination revealed typical microstructure of each tested composite with various ingredient ratios and particulate/fiber fillers size and shape in composite matrix *(Figure 4*). This proposed a justification for dissimilar performance among tested composites.

DISCUSSION

Based on the results of the current study, tested surface characteristics were affected by the composite type, driving us to reject the null hypothesis. In our previous research, we presented a new SFRC CAD/CAM blocks having promising performance related to mechanical properties and fracture-behaviour.[13] Despite that, no information about surface related properties are available. Data showed that inclusion of short fibers with CAD/CAM composite block did not negatively influence the surface characteristics of composite in comparison with different commercial composites.

In this study, the experimental SFRC CAD/CAM block and commercial flowable SFRC (everX Flow) consisted of the same quantity of discontinuous short glass fibres with a diameter of 6 µm and length in range of 200-300 µm.[14] Though, the flowable SFRC (everX Flow) has earlier been reported to have comparable surface gloss values to conventional and fluoride-releasing composites.[15] Interestingly, the experimental SFRC block showed better surface properties than commercial SFRC except for wear test, they behaved similar. This could be attributed to the difference in particulate filler inclusion, which resulted in improved surface properties of experimental SFRC CAD/CAM composite *(Table 1*).

Superior gloss for a composite offers a natural, aesthetic look to a restoration. Based on ISO semigloss surfaces like composites have to be evaluated with 60° angle of lighting, which was utilized in the current study. This used to be located nearer to the viewpoint from which regular person will look at the surface.[16] The large amount of included nanosized particles

Table 3. Major elemental compositions of investigated composites determined with EDS analysis

Materials	Weight %
TEMP PRINT	C 40.9%, O 36.2%, N 11.2%, Si 11.4%, Ba 0.3%
GR-17	C 46.1%, O 34.2%, N 7.8%, Al 1%, Si 6.3%, Ba 4.6%
Essentia U	C 17.7%, O 33.8%, N 6.9%, Al 3.5%, Si 19.1%, Ba 19%
Gradia Plus	C 18.4%, O 32.4%, N 8.1%, F 6.4%, Al 9.1%, Si 9.6%, Sr 15.5%, Ba 0.5%
Cerasmart 270	C 16.5%, O 33.5%, Al 4%, Si 24%, Ba 21.5%, Na 0.5%
everX Flow	C 25.9%, O 33.9%, N 5.3%, Mg 0.1%, Al 3.5%, Si 16.7%, Ca 3.3%
SFRC Block	C 15.5%, O 35.5%, N 8%, Al 4%, Si 18.5%, Ba 15%, Ca 3.5%

(av. Ø 20 nm) in Cerasmart 270 leads to higher surface gloss values than did most of the other evaluated composites *(Table 2)*. This finding in line with earlier studies on the impact of filler size on the gloss of composites.[17,18] Hence, certain other investigations have proven that elements like monomer type, refraction index and degree of monomer conversion can additionally have an effect on the gloss of composites.[19,20] Corresponding to the American Dental Association (ADA) professional product review, 40–60 GU used to be recognized as a normally preferred gloss based totally on observations from specialist panelist.[21] Based on this, all tested composites exhibited successful gloss values except for GR-17 composite which intended for interim clinical use.

The wear of dental composite is a complicated procedure including fatigue, along with abrasive, adhesive and erosive elements.[22] The two-body wear test has been designed to mimic clinical wear and several researchers have utilized, even though a significant differences in the outcomes have been observed even with the same material and testing technique.[22] In this study, SFRCs (CAD/CAM block and everX Flow) revealed comparable wear depth values than some evaluated commercial composites *(Figure 3)*. Thereby, microfiber filler loading were not deteriorating the wear of the SFRCs.[23] Although TEMP PRINT (3D-printed) composites have lower particulate filler content than Cerasmart 270, both showed the lowest wear depth values among other tested composites *(Figure 3)*. This should be owing partly to the resiliency of the composite matrix which provide some shock-absorbing ability.[24] On the other hands, the low wear depth values of Cerasmart 270 showed good connection with the surface microhardness results. However, the rest of results did not show a correlation between the surface microhardness and the two-body wear, which is in line with some previous findings.[25,26]

According to the surface microhardness outcome of this study, the degree of surface microhardness appears to be associated to the filler loading of the composite material and curing efficiency. Visible light with adequate intensity and wavelength and for a proper curing time are critical for sufficient polymerisation of photo-polymerised composites.[27] Accordingly, CAD/CAM composites showed higher surface microhardness values than other 3D-printed and manually-made composites *(Table 2)*.

As recommended by Habib *et al.*, and Mörmann *et al.*, in the present study we assessed the surface roughness of polished composites pre and post wearing test to homogenize the specimens.[28,29] It was noted that the average surface roughness of all tested composites before wearing was much lower than after wearing test *(Table 2)*. Such variation could be attributed to the alteration of surface topography due to loss of surface filler particles and abrasion of resin matrix *(Figure 2)*. The polished surfaces of SFRC CAD/CAM blocks had been surprisingly smooth, comparable to that of particulate filled composites *(Table 2)*. The microfibers protrusion was not recognized and in lieu of the fibers being pulled out to create a pitted surface, the fibers have been polished down with the resin matrix *(Figure 4)*. According to Bayne and his colleagues, the filler loading is not as critical as the pattern of fillers scattering inside the matrix and the inter-particle spacing of filler particles has an important role in the protection of composite surface30. In some other research, the quality of surface characteristics of the nanofilled composite (av. Ø 175 nm) before and after abrasion test proposed that smaller filler particles might be beneficial for keeping excellent surface characteristics after being exposed to oral environment.[31]

Several authors in the literature demonstrated the link between surface gloss and surface roughness.[32-34] The lower the surface roughness the higher the surface gloss. Heintze *et al.*, stated that the surface gloss enhanced continuously throughout the polishing process.[32] However, some authors showed that the enhancement of surface roughness had not been equal to the enhancement of surface gloss, and varied from composite to composite.[35,36]

In this investigation, the effect of long-term water storage on the surface characteristics of tested composites was not studied. However, it has been previously reported that resin containing restorative materials are prone to hydrolytic degradation as an effect of water on the silane interface as well as by softening and weakening of the matrix itself due to an absorption of water by resin components.[37] Another limitation, a group of composites polished with chair-side polishing protocol is missing and this will be evaluated in the near future. To mimic more clinical environment and to have a perfect view of material behavior under clinical conditions, brushing abrasion and thermal aging should also be taken into consideration. Moreover, aesthetic properties and color/gloss stability of the experimental SFRC CAD/CAM blocks need to be evaluated in the future.

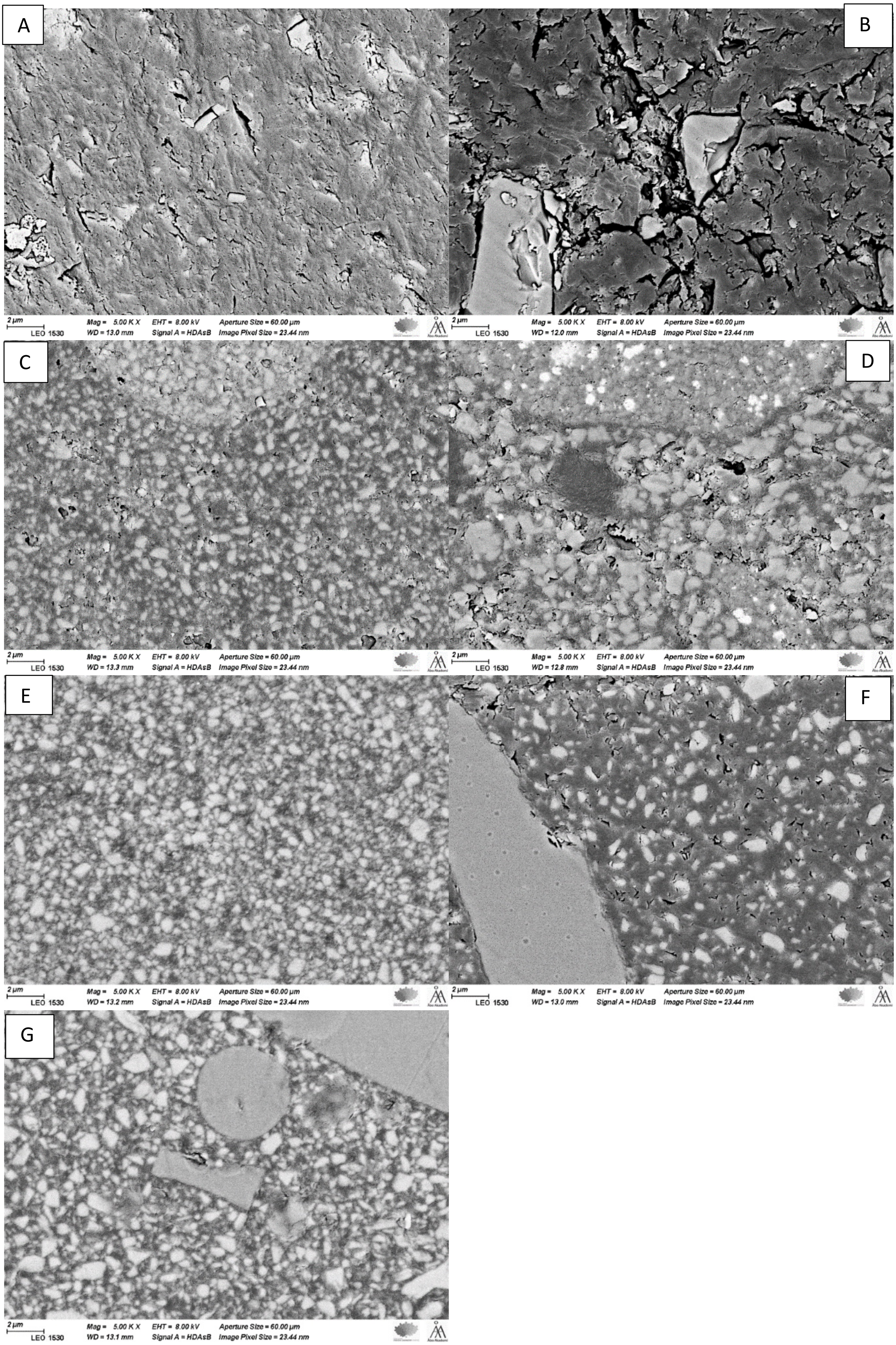

Figure 4: SEM photomicrographs (magnification: 5000 x) of polished surface of investigated materials (scale bar= 2 µm). (A) TEMP PRINT; (B) GR-17; (C) Essentia U; (D) Gradia Plus; (E) Cerasmart 270; (F) everX Flow; (G) SFRC Block.

CONCLUSION

Within the limitations of this study, the following conclusions can be drawn:

1. Incorporation of short fibers to the composite of the CAD/CAM block did not negatively influence the surface microhardness, roughness, gloss and wear of composite.
2. Temp PRINT (3D-printed) and Cerasmart 270 (CAD/CAM) presented the lowest values of wear depth compared with all tested composites.
3. Cerasmart 270 showed superior surface microhardness, roughness and gloss than the other tested composites.

ACKNOWLEDGMENTS

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MANUFACTURER DETAILS

- SpeedMixer DAC 400.1, Hauschild GmbH, Germany
- Rotopol-1; Struers, Copenhagen, Denmark
- Mitutoyo Corp, Kanagawa, Japan
- Quantrex 90, L&R Ultrasonics, Kearny, NJ, USA
- Elipar TM S10, 3M ESPE, Seefeld, Germany
- Targis Power, Ivoclar Vivadent AG, Liechtenstein
- Asiga Max UV, Asiga, Sydney, Australia
- Struers, Glasgow, Scotland
- CS-4.2, SD Mechatronik, Feldkirchen-Westerham, Germany
- Bruker Nano GmbH, Berlin, Germany
- Struers, Duramin, Copenhagen, Denmark
- Zehntner-Glossmeter, GmbH Testing Instruments, Germany
- SEM, LEO, Oberkochen, Germany
- BAL-TEC SCD 050 Sputter Coater, Balzers, Liechtenstein
- SPSS, IBM Corp. NY, USA
- Gradia Plus, GC, Tokyo, Japan
- CERASMART 270, GC, Tokyo, Japan
- Essentia Universal, GC, Tokyo, Japan
- TEMP PRINT medium, GC, Tokyo, Japan
- GR-17 temporary, Pro3dure Medical, Iserlohn, Germany
- everX Flow, GC, Tokyo, Japan

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