EFFECT OF WEAR ON HARDNESS OF 3D PRINTED AND CAD-CAM MILLED RESIN-BASED RESTORATIVE MATERIALS

(IN VITRO STUDY)

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ABSTRACT

BACKGROUND: Recently introduced permanent fixed dental restorations using milled and 3D printed materials. They must exhibit biocompatibility and possess sufficient mechanical strength to endure mastication. Currently, no material meets these requirements.

PURPOSE: To assess the impact of wear on resin-based restorative materials produced through 3D printing and milling.

MATERIALS AND METHODS: Twenty bar-shaped specimens (15 mm x 4 mm x 1.5 mm) were divided into two groups: 3D printed Flexcera Smile Ultra+ permanent resin (n=10) and milled BRILLIANT Crios (n=10). All groups finished with composite polishing kits. Artificial toothbrushing machine wear was tested. A weight loss test was used to measure specimen wear. Vickers hardness test was used to quantify the surface microhardness of additive manufactured and milled resin specimens and assign a Vickers hardness number value. Wear and hardness data were analyzed using one-way ANOVA and Tukey's post hoc test.

RESULTS: All materials exhibited wear reduction following the intervention, whereas Flexcera Smile Ultra+ showed the least and BRILLIANT Crios the highest. Significant changes in hardness were found between all groups before and after intervention ($p < 0.0001$). This demonstrated that the intervention reduced hardness in all materials, with varying degrees of change.

CONCLUSION: Based on their properties, artificial toothbrushing affected each material differently. 3D-printed resin had the lowest wear and hardness value. The highest wear and hardness were found in milled resin.

KEYWORDS: Wear, Flexcera, BRILLIANT Crios, Vickers hardness number.

RUNNING TITLE: Effect of wear on mechanical properties

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INTRODUCTION

Digital advancements have brought about a significant transformation in the field of prosthodontics. Specifically, the incorporation of computer-aided design and computer-aided manufacturing systems has become a regular part of daily practice. These systems utilize both subtractive manufacturing and additive manufacturing techniques. Among the various additive manufacturing methods available, digital light processing has gained widespread usage in dental procedures. Irrespective of the specific technology employed, additive manufacturing has significantly improved production processes by enabling economical manufacturing with minimal raw material and the capability to fabricate products with intricate and complicated parameters (1).

Digital light processing technology microsystem is composed of a rectangular configuration of mirrors known as a digital microreflector device. Each mirror corresponds to a single pixel and the resolution of the displayed image relies on the quantity of mirrors present. The angles of the microreflectors can be individually adjusted. The light emitted by the light source is bent by the micromirror and then projected onto the intended printing surface as a single pixel. In comparison to the sequential scanning of layers using lasers in stereolithography technology, digital light processing offers the advantage of constructing the entire layer through a single laser exposure. Since each layer is independently constructed without considering the shape or number of pixels in the respective layer, this approach reduces construction time (2).

Two resin materials, one 3D printed resin and one resin composite computer-aided design and computer-aided manufacturing block, were utilized for this study.

Flexcera Smile Ultra+ is a unique dental material. Unlike most resins on the market, Flexcera Smile

Ultra+ was specifically developed with improved moisture resistance, high work fracture toughness, and ceramic nanoparticles for enhanced esthetics and color stability (3).

BRILLIANT Crios is a type of material that can be used for subtractive manufacturing. It is a reinforced composite resin containing amorphous silica and glass ceramic fillers, which are embedded in a cross-linked methacrylate matrix. BRILLIANT Crios has a modulus of elasticity measuring 10.3 GPa (4).

Wear resistance is an important physical property in dentistry as it can predict the durability and longevity of different restorative materials during function (5). Excessive wear may reduce the vertical dimension of occlusion causing premature contact in the anterior segment, reduction in the masticatory efficiency, muscles of mastication fatigue, and impaired esthetics (6).

The objective of this in vitro study was to investigate the wear properties and hardness of computer-aided design and computer-aided manufacturing restorative materials, regardless of whether they were produced through 3D printing or milling. The study sought to examine various null hypotheses, including:

There would be no variation in the wear characteristics of the computer-aided design and computer-aided manufacturing restorative materials when exposed to higher loads, irrespective of whether they were 3D printed or milled,

There would be no variation in the hardness of the computer-aided design and computer-aided manufacturing restorative materials before and after simulated toothbrushing, irrespective of whether they were 3D printed or milled (6).

MATERIALS AND METHODS

This in vitro study was approved by the Research Ethics Committee at the Faculty of Dentistry, Alexandria University IORG 0008839. To create ten bar-shaped specimens with specific dimensions (15×4×1.5 mm) as showed in Figure (1), a STL file was initially designed using Blender software 3.2 (Bender Institute, Netherlands). The final STL file was then printed using digital light processing technology as showed in Figure (2), employing a 3D printer (Phrozen Sonic Mini 4K, Xiangshan Dist., Hsinchu city 30091, Taiwan). These composite resins were printed in a vertical orientation on the platform, using a liquid resin material called Flexcera smile ultra +. Once the printing is completed, the ten 3D printed specimens were carefully separated from the build platform using a spatula. Subsequently, a cleaning process was carried out following the manufacturer's recommendation in two steps with ethanol (96 %) using an unheated ultrasonic bath (Codyson CD-4820, Shenzhen, China). First, for 3 min in a reusable ethanol solution (96 %) then cleaned carefully for another 2 min in a freshly used ethanol (96 %) solution. Finally, the 3D printed specimens were removed from the ethanol bath and sprayed with additional ethanol (96 %) to totally get rid of any remaining resin residue. Following the cleaning process, the 3D printed specimens were dried using compressed air under an extraction unit. To achieve the desired mechanical properties and ensure full polymer conversion while reducing residual monomers, the printing specimens underwent post-curing for 45 min, then left to cool for 3-5 min in an ultraviolet light curing device (Brelux power unit 2 post-curing unit, Chesterfield UK). Finally, all supporting structures of the final printed products were removed using a cutting wheel.

To create ten bar-shaped specimens with specified dimensions (15 mm×4 mm×1.5 mm), BRILLIANT Crios blocks (Coltène, Whaledent A.G. Altstatten, Switzerland) were crafted using a high-precision electric saw (Isomet 4000, microsaw, Buehler Ltd, USA) equipped with a water-cooling system utilizing diamond disc (Buehler instrument, USA) with a thickness of 0.6 mm. Subsequently, the specimens underwent refinement with wet silicon carbide (400 ISO/FEPA, average grain size 35 µm) until achieving the specified dimensions of 15 mm in length, 4 mm in width, and 1.5 mm in thickness using a digital caliper (IP54, Qfun, China) to verify the dimensions in accordance with ISO 6872:2015 standards with accuracy 0.01 mm. The prepared specimens were stored in a dry condition at room temperature. (7).

All specimens (n=20) were polished using a composite polishing kit (EVE Composoft, Keltern, Germany) (8), with a contra angled low speed handpiece (W&H, Bürmoos, Austria) connected to a micromotor. This polishing process aimed to achieve a perfectly smooth surface on all specimens from the test groups. Subsequently, all specimens were re-immersed in an ultrasonic cleaner (Codyson CD-4820, Shenzhen, China) for the cleaning procedure. The cleaner was filled with 96% ethanol.

After cleaning, they were dried using absorbent paper and air before being weighed (9).

The specimens were immersed in distilled water and stored in an incubator at 37.5 °C for 1 week (9). The specimens were weighed first before the artificial toothbrushing using a sensitive electronic balance (RADWAG laboratory analytical balance, Radom, Poland).

For all specimens (n=20), a two-body wear test was conducted using custom made toothbrushing wear machine. Mechanical brushing was done for a total of 10000 strokes: simulating one year of clinical use (9).

A vertical force of 2.0 N (equivalent to 200 grams) was exerted using a toothbrush (Colgate Twister; Colgate-Palmolive, São Paulo, SP, Brazil). A toothpaste slurry was created by combining 250 grams of Colgate Total toothpaste (Colgate Total, Colgate-Palmolive; relative dentin abrasion = 70) with 1 liter of distilled water, following the ISO 14569-1:2007 standard. The slurry was renewed after every 5,000 cycles. The specimens underwent a cleaning process using an ultrasonic cleaner. Subsequently, specimens were rinsed with water and dried upon completion of the cycle (10). The magnitude of wear was quantitatively evaluated by measuring the weight loss (10). Therefore, all the examined specimens underwent weight reduction evaluation prior to and following the wear test. Prior to and following the wear test, the specimens were measured using an electronic balance.

The data for wear assessment was collected as follows:

Mean value weight change: weight before wear test- weight after wear test.

Weight loss percent = (w1-wf/ w1) X 100.

Where W_1= initial weight before wear test in (mg).

Wf = final weight after wear test in (mg).

Vickers hardness number was determined of the material before and after artificial toothbrushing test using a microhardness indentation device (HST-HV1000Z, Jinan 250012, PR China) in accordance with the ISO 14233:2003 standard.Each of the two groups consisted of 5 subjects (total n = 10). For each specimen, three indentations were created with approximately 3 mm spacing between them. To calculate the Vickers hardness number, the average lengths of the two diagonal lines visible within each indentation were measured. The indentations were made using a diamond pyramid micro-indenter with a 136° angle between the opposing faces under a 1.961 newtons (N) load for 15 seconds to determine the Vickers hardness number value. The average value of these three measurements was used to represent the Vickers hardness number of the material.

The Vickers hardness number (H) was calculated using Equation 2: H = 1.961 F / D^2 in this equation

F represents the applied load in newtons (N).

D is the area of the tip of the indenter in square millimeters (mm^2) (10).

Normality was checked using *Shapiro Wilk test* and *Q-Q plots*. Wear and Hardness were normally distributed while percent change in all measured parameters. Data values were summarized using mean, standard deviation (SD), minimum and maximum values. Percent change was calculated according to the following formula:

$$\frac{Values\ after\ toothbrushing - Values\ before\ toothbrushing}{Values\ before\ toothbrushing} x\ 100$$

Differences in wear and hardness were analyzed using *One Way ANOVA* and followed by *Tukey's post hoc test* with Bonferroni correction while changes before and after artificial toothbrushing were assessed using *Paired t test*. *Kruskal Wallis test* with *Dunn's post hoc test* with Bonferroni adjustment was employed to analyze percent change values between groups. All tests were two tailed and the significance level was set at p value<0.05. IBM SPSS version 23, for Windows, Armonk, NY, USA was used for data analysis.

Figure (1): 3D printed specimens printed specimen.

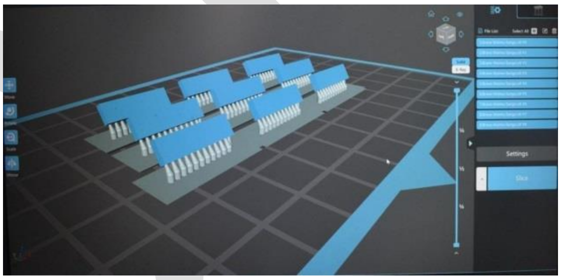

Figure (2): Pre-processing before 3D printing.

RESULTS

Wear test

The study assessed the baseline weight of the BRILLIANT Crios, and Flexcera Smile Ultra+ groups before the intervention, as well as the wear experienced after the intervention. (Table 1). The mean ±SD baseline weight before intervention was 0.192 ± 0.005 for BRILLIANT Crios, and 0.114 ± 0.007 for Flexcera Smile Ultra+. Post-intervention, mean ±SD wear values declined to 0.189 ± 0.005 for BRILLIANT Crios, and 0.113 ± 0.007 for Flexcera Smile Ultra+. Using One Way Analysis of Variance ANOVA, significant wear differences were found between groups before and after the intervention ($p < 0.0001$).

Additionally, paired t tests indicated significant reductions in wear within each group following the intervention: Flexcera Smile Ultra+ ($p < 0.0001$) and BRILLIANT Crios ($p = 0.006$). Pairwise comparisons further confirmed significant differences in wear between the study groups ($p < 0.0001$) both before and after the intervention (Table 2).

These findings highlighted that all materials experienced a reduction in wear following the intervention.

Hardness test

The study evaluated BRILLIANT Crios, and Flexcera Smile Ultra+ groups hardness before and after intervention (Table 3). Before intervention, BRILLIANT Crios had 71.43 ± 4.08, and Flexcera Smile Ultra+ had 16.73 ± 0.70. After intervention, these values changed significantly, with BRILLIANT Crios to 59.10 ± 3.08, and Flexcera Smile Ultra+ to 12.20 ± 1.74.

Statistical analyses using One-way ANOVA (Test1) revealed significant hardness differences between groups before and after intervention (p < 0.0001). Paired t tests (Test2) revealed significant decrease in hardness within each group post-intervention of the study groups (p < 0.0001).

Pairwise comparisons confirmed significant differences in hardness between all groups both before and after intervention (p < 0.0001). This demonstrated that the intervention effectively decreased the level of hardness in all materials, with varying degrees of change (Table 2). The material with the least amount of change was BRILLIANT Crios and Flexcera Smile Ultra+ showed the highest level of change.

Table (1): Comparison between the baseline weight before the intervention and the wear exhibited after the intervention among the two groups.

		Brilliant Crios (n=10)	**Flexcera Smile Ultra+ (n=10)**	**Test^1 (*p* value)**
Before	Mean ±SD	0.192 ±0.005	0.114 ±0.007	301.424 (<0.0001*)
	Median	0.194	0.116	
	Min – Max	0.182 – 0.197	0.102 – 0.120	
After	Mean ±SD	0.189 ±0.005	0.113 ±0.007	282.409 (<0.0001*)
	Median	0.192	0.115	
	Min – Max	0.180 – 0.195	0.101 – 0.119	
Test^2 (*p* value)		3.621 (0.006*)	10.500 (<0.0001*)	

*Statistically significant difference at *p* value<0.05, Test^1: One Way Analysis of Variance, Test^2: Paired t test

Table (2): Two Pairwise comparisons between baseline weight/hardness before the intervention and the wear/hardness exhibited after the intervention among the two groups.

Groups	Compared to	*p* value	
		Before	After
Brilliant Crios regarding wear test	Flexcera Smile Ultra+ for wear test	<0.0001*	<0.0001*
Brilliant Crios regarding hardness test	Flexcera Smile Ultra+ for hardness test	<0.0001*	<0.0001*

*Statistically significant difference at *p* value<0.05

Table (3): Comparison of hardness among the study groups before and after intervention.

		Brilliant Crios (n=10)	**Flexcera Smile Ultra+ (n=10)**	**Test[1] (*p* value)**
Before	Mean ±SD	71.43 ±4.08	16.73 ±0.70	679.044 (<0.0001*)
	Median	74.00	16.60	
	Min – Max	66.00 – 74.30	16.00 – 17.00	
After	Mean ±SD	59.10 ±3.08	12.20 ±1.74	713.336 (<0.0001*)
	Median	61.00	12.00	
	Min – Max	55.00 – 61.30	10.30 – 14.30	
Test[2] (*p* value)		37.00 (<0.0001*)	13.071 (<0.0001*)	

*Statistically significant difference at *p* value<0.05, Test[1]: One Way Analysis of Variance, Test[2]: Paired t test

DISCUSSION

Recently, long-term dental restorations have employed dental resin-based materials that are produced by additive and subtractive manufacturing techniques. Dental crown resins need to have strong mechanical properties that can endure the forces applied during mastication while still being biocompatible. At present, there is no substance that meets these criteria (11). This study assessed the wear and microhardness of specific resin composites commonly used for milling and 3D printing of permanent dental restorations. The evaluation was conducted before and after subjecting the composites to artificial toothbrushing. The null hypothesis regarding wear and hardness was rejected.

The 3D printed resin was selected. Flexcera Smile Ultra+. The intention of this material is to provide resistance to moisture, have a refractive index that matches natural teeth, possess high work fracture toughness, and contain ceramic nanoparticles for improved esthetics (3). Renne (3) attributed this phenomenon to the unique oligomer structure of the substance, which is characterized by long chains chemistry. Compared to subtractive manufacturing, additive manufacturing can build larger objects, produce accurate features, and reduce material waste (8). BRILLIANT Crios is a cross-linked methacrylate matrix reinforced composite resin with amorphous silica and glass ceramic fillers, 10.3 GPa (4), modulus of elasticity, 70.7% by weight and 51.5% by volume filling, and 198 MPa bending resistance. For restorative stress reduction and fracture prevention, a dentine-like modulus of elasticity was recommended (11). Dentine-like modulus of elasticity makes BRILLIANT Crios excellent for dental restorations because of shock absorption. (12). This study studied computer-aided design and computer-aided manufacturing ceramic-filled resin material, which reduced chair time, improved esthetics, and strengthened restorations (13).

Wear test

The comparison of wear between research groups before and after the intervention showed a reduction in wear after the intervention. Notably, Flexcera Smile Ultra+ consistently exhibited the lowest wear, whereas BRILLIANT Crios had the highest wear. Two opinions might be attributed to the filler morphology which states that: The reported results can be linked to the filler shape in two ways. The first is the introduction of spherical silica particle fillers, as seen in Flexcera Smile Ultra+, which improves wear resistance, which is consistent with previous study conducted by Xing et al., (14) The second reason is that BRILLIANT Crios fillers are angular in shape, and object morphology plays an important role in two-body abrasion (15). Objects with angular protrusions are more prone to wear faster than spherical ones.

An alternative explanation for the observed results could be ascribed to the combinations of monomers. BRILLIANT Crios consists of Bis-GMA (Bisphenol A-glycidyl methacrylate), Bis-EMA (Ethoxylated bisphenol A-glycol dimethacrylate), TEGDMA (Triethylene glycol dimethacrylate), barium glass, and 70% silica particles. Nguyen et al., (16) found that using only (Urethane dimethacrylate) UDMA resulted in higher mechanical properties when the fillers remained constant, as opposed to using a mixture of UDMA and TEGDMA in varying proportions. In addition, Szczesio et al., (17) proposed that the combination of UDMA/Bis-EMA/TEGDMA monomers have superior mechanical properties in comparison to UDMA/TEGDMA.

In addition, Nguyen et al., (18) conducted research to assess the mechanical characteristics of computer-aided design and computer-aided manufacturing resin composite blocks utilizing a BIS-GMA polymer matrix, in comparison to experimental resin composite blocks that rely on a UDMA matrix. The investigation revealed that the UDMA block had improved mechanical properties when compared to the BIS-GMA block.

The presence of a sintered network was responsible for the significant increase in strength and hardness. In addition, the UDMA matrix facilitated the formation of a sturdier and more compact network, leading to enhanced mechanical characteristics.

Various research findings validate that wear is a multifaceted phenomenon, where outcomes are heavily influenced by the design and settings of the test. Conducting wear experiments, such as pin-on-block, pin-on-disc, three body wear, and toothbrush simulation, under various stresses, sliding conditions, and environmental factors (such as water or food bolus), might yield diverse outcomes (19). The variations in average wear could potentially be accounted for by distinct material characteristics, such as hardness or modulus of elasticity. The absorption of water into the polymer matrix can potentially cause the resin composite to become softer (20) or cause the silane agent to undergo hydrolysis (21), leading to a decrease in the wear resistance of the resin composites.

Another explanation for the findings might be attributed to the comparable strength of the polymeric matrix and inorganic fillers resulted in a homogenous wear across the material surface, thereby increasing the wear reduction rate of the BRILLIANT Crios block.

This study contrasted with the findings of Arafa et al. (6), who found significant weight reduction in materials produced using 3D printing and milling. There was no discernible disparity in weight across the groups. Significant weight variation was seen in the 3D printed materials. This outcome might be ascribed to the inclusion of filler materials, the implementation of standardized manufacturing procedures for computer-aided design and computer-aided manufacturing blocks at specific temperature and pressure conditions, or the existence of an oxygen inhibiting layer in 3D printed resin materials.

Sumino et al. (22) discovered another inconsistency in their analysis, proposing that the wear characteristics of resin composites may be attributed to the specific type and size of distributed fillers. It was hypothesized that resin composites containing bigger filler particles would exhibit greater wear, but those with smaller filler particles might be densely packed, resulting in less exposure of the polymer matrix to wear and therefore reduced wear.

Hardness test

Hardness variations between research groups before and after the intervention exhibited a reduction in hardness following the intervention. Flexcera Smile Ultra+ consistently showed the lowest Vickers hardness number, whereas BRILLIANT Crios consistently showed the highest Vickers hardness number. The results of this study demonstrate that the intervention is efficient in lowering the hardness of all materials, with varied degrees of change being detected across the materials that were examined.

The variations in surface hardness among materials can potentially be attributed to the composition of the components. Furthermore, the presence of inorganic fillers and the application of high temperatures during the polymerization process of computer-aided design and computer-aided manufacturing resins may contribute to the enhancement of certain mechanical qualities, such as surface hardness (23). This outcome could be linked to the storage of a wet or moist environment, which could cause the absorption of water by composite resins. Consequently, this could contribute to the breakdown of the composite resins and the decline of their mechanical qualities (24).

The findings of this investigation were compatible with the research conducted by Grzebieluch et al., (7) which demonstrated a notable disparity in hardness. These results align with the findings reported by Ling et al., (25) Grzebieluch's findings indicated that the most challenging materials in this particular set are milled blocks, mostly due to their structural characteristics, as per his assessment. A strong link was found between the amount of filler and the microhardness, despite variations in material structure. Ling et al., (25) and Mirică et al., (26) observed a comparable connection. The hardness of a material is also correlated with its susceptibility to abrasion, and materials with low hardness are more prone to wear (27) which was incompatible with our results. Therefore, it can be inferred that the deterioration of printed material will occur at a faster rate compared to milled materials.

This finding aligns with other studies indicating a positive correlation between filler content and hardness reported by McCabe et al (28) Son et al (29) Loyaga-Rendon et al (30).

The findings of this investigation disagreed with those of Al-Haj et al., (31) who demonstrated that the hardness of 3D printed resins remained unchanged following intervention. Grzebieluch et al., (7) found that the microhardness values of milled blocks were much higher than the values of other materials examined. Nevertheless, no statistically significant disparities were seen in the microhardness measurements of 3D printed resin-based materials.

According to Temizci et al., (32) the microhardness values of the milled and 3D printed materials were found to have a statistically significant difference. The study's findings indicated that the milled blocks exhibited the highest Vickers hardness number value, but the 3D printed materials demonstrated the lowest Vickers hardness number value prior to undergoing thermocycling. They provided an explanation that suggested it may be attributed to the composition.

Goujat et al., (33) and Kim et al., (34) evaluated the surface hardness of computer-aided design and computer-aided manufacturing materials and 3D printed resins. The results showed that computer-aided design and computer-aided manufacturing materials had a higher surface hardness compared to 3D printed resin, which is consistent with the trend seen in this study. Surface hardness studies conducted after simulating toothbrushing showed a modest elevation in the surface hardness of 3D printed resins, which can be regarded as a notable finding.

CONCLUSION

The results of this investigation lead to the following conclusion:

Each material was affected by artificial toothbrushing

Flexcera Smile Ultra+ consistently showed the lowest wear, and BRILLIANT Crios the highest.

The highest value in hardness was shown in BRILLIANT Crios and the lowest value was presented in Flexcera smile ultra+.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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