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FRACTURE RESISTANCE AND MARGINAL FIT OF CERAMIC REINFORCED RESIN CROWNS FABRICATED BY ADDITIVE MANUFACTURING (IN VITRO STUDY)

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ABSTRACT

BACKGROUND: The fit and strength of the additively manufactured restorations have not been thoroughly investigated.

OBJECTIVES: this study was conducted to evaluate the marginal fit and fracture resistance of hybrid ceramic crowns fabricated by additive and subtractive manufacturing.

MATERIAL AND METHODS: A Maxillary first premolar typodont tooth was prepared to receive a hybrid ceramic full crown. The prepared tooth was duplicated into 18 epoxy resin dies that were divided into 3 groups (n=6): subtractive manufacturing of Polymer network infiltrated ceramic (Vita enamic) (VE) crowns, the other 2 groups were additively manufactured using ceramic-reinforced resin Flexcera smile ultra+ (Flex) and Varseosmile crown plus (VS) respectively. Intraoral scanner was used to scan all epoxy resin dies. A dental CAD program was used to make a standardized crowns design. VE was fabricated using a dental milling machine, while Flex and VS were fabricated using a 3D resin printer. Marginal gap was measured before cementation and after cementation and thermo-mechanical aging. All specimens were subjected to load till failure using a universal testing machine, followed by failure mode analysis. Statistical analysis was performed using Kruskal Wallis and Wilcoxon signed ranks tests.

RESULTS: VITA Enamic and Varseosmile had statistically significant higher fracture resistance compared to Flexcera. VITA Enamic had statistically significant largest marginal gap than both Flexcera and Varseosmile. Thermomechanical aging significantly increased the marginal gap of all groups.

CONCLUSIONS: VITA Enamic and Varseosmile demonstrated significantly better fracture resistance compared to Flexcera. Printed crowns showed less marginal gap than milled crowns.

KEYWORDS: Hybrid ceramics, Additive manufacturing, Subtractive manufacturing, Marginal fit, Fracture resistance.

RUNNING TITLE: Ceramic-reinforced resin crowns.

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INTRODUCTION

Computer-aided design and computer-aided manufacturing (CAD-CAM) technology has been used in dentistry for decades to fabricate prosthetics ranging from single crowns to complete mouth restorations. Dental restorations have been long associated with subtractive milling of solid highly compact material shaped as blocks or blanks. Milling is performed by removing substantial material around and inside the intaglio of the restoration (1).

Various materials are available as solid blocks to be milled using dental milling machines including zirconia, glass ceramics and hybrid ceramics. Recently, hybrid ceramics gained popularity for making single tooth restorations as it contains high percentage of ceramic fillers or network in resin matrix, which provides not only high strength in comparison to conventional composites but also cushion effect that distributes and absorbs stresses (2).

Hybrid ceramics are considered highly machinable materials due to their low hardness resulting in less resistance to milling tools (2). This high machinability provides accurate restorations at the fitting surface and margins improving internal and marginal fit in comparison to glass ceramics. However, milling processes are generally affected by various factors including the quality, size and sharpness of milling tools, cooling system and milling strategy (3).

Recently, additive manufacturing has been introduced to the dental market to fabricate dental restorations. The process involves sequential layering of the material to fabricate a final restoration. This method allows the clinician to

reduce the fabrication expenses as additive manufacturing results in less material waste (4).
Additive manufacturing of hybrid ceramic materials has recently been introduced to make permanent single tooth restorations using 3D printed materials that are highly filled photopolymerized viscous liquid resin. These materials are additively manufactured using direct light projection or comparable methods to make photopolymerization of the material in sequential layers (5). In comparison to subtractive manufacturing, additive manufacturing produces pieces with complex geometries and narrow areas as in the intaglio of the crown incisally or occlusally and at the thin margin areas (5).
The filler content, type and form of the hybrid ceramic materials are the main determinants of strength of the materials. In literature, many studies have investigated the strength and fracture resistance of different hybrid ceramics, and conflicting results were found. Zimmermann et al., (6) found that 3D-printed composite resins achieved similar fracture loads to Vita Enamic CAD-CAM blocks, but these results may vary depending on the specific 3D-printing material and crown thickness. Corbani et al., (7) reported that the 3D printed materials showed the highest values for fracture resistance compared with the milled group within the three tested thicknesses. On the other hand, Çakmak et al., (8) reported that Crowns fabricated using subtractive manufacturing form polymer-infiltrated ceramic network material had the highest fracture resistance compared to additively manufactured materials.
Data on fracture strength and marginal fit of 3D printed restorations relative to milled restorations are limited and require further research. Hence, the current study aims to investigate the fracture resistance and marginal fit of 3D printed restorations. Null hypotheses of this study were that no difference would be found in the marginal fit and fracture resistance of 3D printed and milled CAD-CAM crowns.

MATERIALS AND METHODS

Chemical compositions, Manufacturer companies, and properties of materials used in the present study are shown in Table (1). A maxillary first premolar typodont tooth was scanned by an intraoral scanner (Omnicam, Dentsply Sirona) to capture the preoperative shape of the tooth. After scanning, the tooth was prepared to receive a hybrid ceramic full crown. The occlusal reduction was 1.5 mm and the preparation dimensions were 6 mm height, 6 mm facio-lingually, and 4 mm mesiodistally at the cervical level, with 1 mm circumferential chamfer finish line and 6 degrees axial taper. The prepared tooth was duplicated into 18 epoxy resin dies by the addition of silicone duplicating material. These dies were divided into three groups (n=6): (1) Vita Enamic CAD-CAM, (2) Flexcera smile ultra+ and (3) VarseoSmile Crown plus.
Each epoxy resin die was scanned by an intraoral scanner (Omnicam Dentsply Sirona) to create the definitive scan. After scanning was completed, standard tessellation language (STL) files were exported to be used for the computer-aided design program. The preoperative and the definitive scans were imported to a dental CAD program (Exocad, Exocad Corp.). The preoperative scan was used to guide the contour of each design to create standardized crowns for all groups. The cement gap was set to be 50 µm, and area at the margins without cement gap was 1.0 mm. All designs were exported as STL files and were imported into either a 3D printing preprocessing and slicing program (Chitubox, CBD-Tech) or computer-aided manufacturing (CAM) (Hyperdent) program according to the material used.
Vita enamic blocks were milled by using a 5-axis milling machine (i250, Imesicore). The milling strategy used a cooling system during the milling of the crowns. After milling, all crowns were checked for initial fit over their corresponding preparation.
For the VS and Flex 3D printed materials, the crowns were printed using DLP 3D printer (Anycubic photon S, Shenzhen, China). The parameters for printing were: 0.05 mm layer height, bottom layer count 8, 6.5 seconds exposure time, 20 seconds bottom exposure and 5 mm lift distance and 60mm/sec lift speed. The crowns were nested in a vertical orientation. On completion of printing, the crowns were separated from the build platform using the spatula and cleaned with ethanol (96%) for 5 minutes using an unheated ultrasonic bath (Anycubic 3D Printer Wash and Cure Machine 2.0) following manufacturing recommendations. The crowns were removed from the supported structure with cutting wheel then checked for fit and finished following the manufacturer's instructions. Post curing of the printed crowns was performed at wavelength 405nm for 2 x 20 minutes exposure cycles in an ultraviolet light curing device (Anycubic 3D Printer Wash and Cure Machine 2.0).
Before and after cementation, all specimens were checked for marginal adaptation using a stereomicroscope (sz1145TR, Olympus, Japan 1990) at 10 X magnification figure (1). Each specimen was placed under the microscope and fixed in position using a specially designed Jig to fix the crowns on the die during marginal gap measurement figure (2).
For the cementation of crowns on their corresponding dies, surface treatment of the fitting surface of restoration was started by etching with 9.5% hydrofluoric acid gel (Itena porcelain etch) for 60 s for VE group and 20 s for VS and Flex, followed by rinsing with distilled water for 30 seconds followed by air drying for 20 s. According to the manufacturer's instruction. Two thin coats of

a silane coupling agent (SILAN-IT Itena) was applied with a micro brush to the etched surface followed by gentle air drying, self-adhesive resin cement (TotalCem Itena) was used to cement all the crowns. Each crown was seated in place by light pressure first followed by static load of 5 kilograms using a custom-made static load device. The excess cement was removed after 2 seconds of initial polymerization followed by photopolymerization for 40 seconds using a light-curing unit (Woodpecker LED-D Wireless, Mident Industrial Co., China).

Mechanical aging of the specimens was done using a chewing simulator (custom made chewing stimulator) (dental biomaterial laboratories, faculty of dentistry, Alexandria University). The teeth with cemented crowns were fixed in a special holder and subjected to 120000 loading cycles at a frequency of 1.5 Hz under a weight of 5 kg (49 N). A metallic rod with a 4 mm diameter round end was used to deliver the load parallel to the long axis of the crowns. After completion of the mechanical aging procedures, all specimens were exposed to 1200 thermocycles (between 55˚C and 5˚C) in an automated custom made thermocycling machine, with dwell times of 30 seconds in each water bath (9).

The specimens were photographed using a camera that was connected to the microscope. The images were transferred to an image analysis software (Image J 1.43U, National Institute of Health, USA) to measure the vertical marginal gap at 6 points for each crown on the buccal and lingual surfaces (at the mesiobuccal, distobuccal, mesiopalatal, and distopalatal points, as well as at the mid-buccal and mid-palatal lines). The measurements of all points in each specimen were recorded in microns before and after cementation and thermo-mechanical aging.

All samples were individually mounted on a computer-controlled universal testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA). All samples were secured to the lower compartment of the testing machine by tightening screws. The fracture test was done by compressive mode of load applied occlusal using a metallic rod with a 6 mm diameter sphere indenter that was attached to the upper compartment of the testing machine at a cross-head speed of 1mm/min. A polyethylene sheet was interposed between the occlusal surface of the specimens and the loading sphere, to achieve homogenous stress distribution and to avoid contact damage with steel indenter. The load at failure manifested by a sharp drop at the load-deflection curve recorded using computer software. The load required to fracture was recorded in Newton. A stereomicroscope was used to investigate the mode of failure of each specimen into crack, chipping or crown fracture figure (3).

Figure (1): Show specimen was fixed in position using a Jig.

Figure (2): Show specimens were checked for marginal adaptation using a stereomicroscope.

Figure (3): Show mode of failure of each specimen into crack, chipping or crown fracture.

Data were analyzed using IBM SPSS for Windows (Version 26.0). Descriptive statistics were calculated as means, medians, standard deviation (SD), and interquartile range (IQR).

Normality was tested using descriptive statistics, Q-Q plots, and Shapiro-Wilk normality test.

Data showed a non-normal distribution, so a non-parametric analysis was adopted.

Comparisons between the three study groups were performed using the Kruskal Wallis test, followed by multiple pairwise comparisons using Bonferroni adjusted significance level.

Comparisons of the marginal gap before and after cementation within each group were performed using the Wilcoxon signed ranks test. The significance level was set at p-value <.05.

RESULTS

Table (2) shows the means of the marginal gap of the three study groups. Before cementation, VITA Enamic had the largest marginal gap (mean ± SD: 21.11 ± 0.73) on the buccal aspect, followed by Flexcera (16.25 ± 0.61) and Varseosmile (15.16 ± 0.85) with significant differences observed (p= .001). Similar trends were observed in the lingual aspect and when averaging both aspects. After cementation and thermo-mechanical aging, the marginal gap increased significantly in all groups, with VITA Enamic having the largest gap (42.30 ± 0.49), followed by Flexcera (33.73 ± 1.16) and Varseosmile (32.89 ± 2.44), with significant differences as well (p = .003). The difference in marginal gap (after-before) was also analyzed, showing that VITA Enamic had the largest increase in gap size, followed by Flexcera and Varseosmile, and these differences were statistically significant (p= .005). The results indicate that there is a significant difference in marginal gap between the three groups (p <.05). Post-hoc comparisons reveal that the difference is statistically significant between VITA Enamic and both Flexcera and Varseosmilesmile, but not between Flaxcera and Varseosmilesmile (p >.05).

Table (3) shows the means of fracture load of VITA Enamic, Flexcera, and Varseosmile. The results show that VITA Enamic and Varseosmilesmile exhibit higher fracture resistance compared to Flexcera (mean ±SD= 603.77 ±52.42, 651.38 ±63.63, and 500.11 ±77.22, respectively). The results indicate that there is a significant difference in fracture resistance between the three groups (p = 0.006). Post-hoc comparisons reveal that the difference is statistically significant between VITA Enamic and Flexcera (p = 0.03) as well as between Flexcera and Varseosmilesmile (p = 0.005), but not between VITA Enamic and Varseosmilesmile (p = 1.00).

Regarding the failure mode, all VE specimens demonstrated non repairable fractures while VS and Flex specimens showed various types of failure modes including chipping (repairable failure mode) 17% , 33% and non-repairable failure mode (catastrophic failure and / or cracking) 25 % cracked and 42% , 58% fractured.

Table 1: Chemical compositions and Manufacturer companies.

Name	Manufacturer	Composition
VITA ENAMIC® HYBRID CERAMIC (VE)	VITA Zahnfabrik, Bad Säckingen, Germany	UDMA, TEGDMA feldspar ceramic enriched with aluminum oxide 86%
Flexcera smile ultra+ (Flex)	EnvisionTEC GmbH Brüsseler Str. Gladbeck Germany Desktop Metal, Inc.	Acrylates, methylacrylates, methacrylate oligomers and monomers, photoinitiators, colorants/dyes, fillers and absorbers.
VarseoSmile Crown plus (Vs)	BEGO Bremer Goldschlägerei Wilh. Herbst GmbH & Co. KG Wilhelm-Herbst-Str. Bremen, Germany	Esterification products of 4,40 isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid, silanized dental glass, methyl benzoylformate, diphenyl(2,4,6-trimethyl-benzoyl) phosphine oxide. Total fillers by weight 30-50%

Table 2: Comparison of the marginal gap between the three study groups

			VITA Enamic	Flexcera	Varseosmile	P value
Before	Average	Mean ±SD	20.77 ±1.03 **a**	16.00 ±0.57 **b**	14.96 ±0.26 **b**	.001*
		Median (IQR)	20.73 (19.86, 21.74)	16.00 (15.40, 16.57)	14.98 (14.84, 15.090	
After	Average	Mean ±SD	42.43 ±0.51 **a**	33.81 ±0.56 **b**	31.86 ±1.31 b	.001*
		Median (IQR)	42.48 (41.93, 42.84)	33.93 (33.42, 34.27)	31.94 (30.66, 33.09)	
P Value 2		Average	0.03*	0.03*	0.03*	

SD: Standard deviation, IQR: Interquartile range, Min: Minimum, Max: Maximum
*statistically significant at p value <0.05
P value 1: Kruskal Wallis test, p value 2: Wilcoxon signed ranks test
a-b: different letters denote statistically significant differences between groups using Bonferroni adjusted significance level.

Table 3: Comparison of fracture resistance between the three study groups

	VITA Enamic	Flexcera	Varseosmile	P value
Mean ±SD	603.77 ±52.42 **a**	500.11 ±77.22 **b**	651.38 ±63.63 **a**	.006*
Median (IQR)	608.92 (549.73, 650.48	516.65 (753.08, 445.27)	637.82 (593.31, 716.00)	
Min– Max	536.73– 670.00	357.31– 575.07	586.36– 743.98	
Post-hoc comparison p value	**VITA Enamic vs. Flexcera: 0.03***			
	VITA Enamic vs. Varseosmile: 1.00			
	Flexcera vs. Varseosmile: 0.005*			

SD: Standard deviation, IQR: Interquartile range, Min: Minimum, Max: Maximum
*statistically significant at p value <0.05
Kruskal Wallis test was used

DISCUSSION

This study investigated marginal fit and fracture resistance of single crowns fabricated by additive and subtractive manufacturing. The null hypothesis was rejected as statistically significant differences were observed between these materials.

Recently Additive manufacturing, produce more precise and accurate restorations than subtractive manufacturing (10). Moreover, Additive manufacturing surpasses subtractive manufacturing methods by offering advantages such as mass production, reduced material waste, time-saving, and the ability to create complex geometries, with undercuts or locations that cannot be produced or are limited by the milling process (10).

To ensure standardization across all crowns, all specimens adhered to consistent design dimensions, thickness, cement gap, and anatomical features. The layer thickness of 3d printing materials were set at 50 µm to enhance fracture resistance and improve interlayer bonding (6). Additionally, using the same 3D printer for printing crowns contributes to reproducibility. Fracture resistance of 3D printed crowns is influenced by printing orientation and layer thickness, vertical printing often results in stronger crowns (11).

Moreover, the elastic modulus of epoxy resin material was reported to be 12.9 GPa which falls within the range of elastic modulus of human dentin (7 -13 GPa). The fracture forces of crown increased as the elastic modulus of the material is within the same range of dentin, So, it was chosen for this study because it provides in dimensional accuracy, surface detail reproduction, strength, and abrasion resistance (12).Marginal adaptation refers to the precise fit of a restoration at its margins, where it interfaces with the prepared tooth structure. An accurate fit is essential for preventing various complications that can compromise the restoration's success (13). Many methods have been used to examine the marginal fit. The most widely used method is the direct microscopic evaluation of gaps along the crown margins. This method is non-invasive, accurate, inexpensive, and reliable (14).

The current study displayed statistically significant differences between VE and additively manufactured groups regarding marginal gap in both timepoints; before cementation and after thermomechanical cycling, whereas VE had largest marginal gaps (p<0.001). This could be attributed to different manufacturing methods, as the milling tools could not be able to produce complex geometries that might be present in the margins and the intaglio of the crowns, affecting the fit of the restorations (15). Moreover, wear of milling tools over time could also affect the marginal and internal adaptation of the restorations. Heat generation due to friction of milling tools with the resin material could also result in distortion of the milled piece affecting the fit of the restoration (16).

This result aligns with the findings of Suksuphan et al., (17) who reported that crowns fabricated using 3D-printed hybrid materials exhibited lower values for the marginal gaps compared to milled crowns. Similarly, Donmez et al., (18) found that 3D-printed implant-supported crowns achieved better marginal adaptation than those produced using traditional milling techniques. Kakinuma et al., (19) observed that 3D-printed resin-composite crowns had fewer marginal gaps compared to milled crowns. Conversely, Mohajeri et al., (20) noted that the 3D-printed temporary crowns group had a larger marginal gap when compared with the milled temporary crowns group, however, this may be related to the use of provisional materials that had differences in chemical composition, filler content and type, and the type of printer (21).

Based on the results of this study, cementation and thermomechanical aging have been found to be deteriorating factors that increased the marginal gaps in resin-based materials. This could be attributed to mismatch in the coefficient of thermal expansion (CTE) between the resin material and epoxy resin die material (22). Also Temperature fluctuations can induce expansion and contraction within restorative materials generating stress leading to formation and growth of cracks. Additionally, differences in CTE of the resin matrix and filler content in response to temperature changes could lead to internal stresses within the material (23). Furthermore, cementation procedures with resin cement affect crown fit, as the viscosity of cement could prevent the complete seating of the crowns (24). Our findings align with studies demonstrated significant differences in the marginal gap discrepancy before and after thermo-mechanical aging of hybrid ceramic restorations fabricated by CAD-CAM (25-27). However, this contradicts Beschnidt and Strub (28) whose research concluded that the aging process did not significantly impact marginal fit. However, this may be related to the use of different materials, preparation design, and using extracted natural teeth rather than epoxy resin dies. Overall the materials in this study were within the clinically acceptable marginal gap limit of 120 µm (29).

The results of this study support that the crowns fabricated from Flexcera material exhibited significantly lower fracture resistance than both VE and VS, leading to the rejection of the second null hypothesis. This difference may be attributed to variations in chemical composition and the type and percentage of filler content, which were not disclosed by the manufacturer. The maximum bite force in the posterior region of male adults can reach up to 520 Newtons (30). Consequently, VE and VS in this study can withstand human biting forces mean ±SD= (603.77 ±52.42, 651.38 ±63.63) respectively.

Few studies have explored the factors influencing the mechanical properties of 3D-printed dental restorations (31). Grzebieluch et al., (31) found that vertically oriented 3D-printed samples exhibited superior mechanical properties. This improvement is attributed to the alignment of the printing layers with the direction of masticatory forces, leading to a more uniform distribution of reinforcing particles within the material.

Our findings are consistent with Zimmermann et al., (6) found no statistically significant differences in the fracture load of CAD-CAM crowns fabricated from 3D printed or milled methods. On the other hand, Corbani et al., (7) reported that the 3D printed group showed the highest values for fracture resistance compared to the milled group across the three tested thicknesses. Çakmak et al., (8) found that polymer-infiltrated ceramic network crowns (subtractive manufacturing) were most resistant to fracture, whereas additively manufactured hybrid resin crowns exhibited the lowest resistance. Similarly, Temizci et al., (32) suggested that current 3D-printed resins may not match the long-term durability and wear resistance of established materials like Vita Enamic. This disagreement might result from differences in chemical composition and manufacturing methods. Additionally Suksuphan et al., (17) demonstrated that CAD/CAM hybrid ceramic crowns exhibit significant fracture resistance, even with a relatively thin occlusal thickness of 0.8 mm. This superior performance is attributed to the material's ability to absorb and dissipate energy through elastic and plastic deformation.

In contrast, the Varseo Smile (VS) material, a 3D-printed resin composite, exhibited the lowest maximum loading force. This lower performance may be linked to its relatively low flexural modulus, which could be influenced by its lower filler content and 3D printing manufacturing process. While a lower filler content might facilitate the 3D printing process, it can compromise the material's mechanical properties, particularly its fracture resistance.

Regarding the failure mode, all VE specimens demonstrated non repairable fractures while VS and Flex specimens showed various types of failure modes including chipping (repairable failure mode) 17% , 33% and non-repairable failure mode (catastrophic failure and / or cracking) 25 % cracked and 42% , 58% fractured. While studies comparing these materials are limited, similar materials were investigated (33).

This study to evaluate marginal fit and fracture resistance for single crowns supported by natural tooth preparation geometry.

Most of the literature discussed marginal fit and fracture resistance for partial coverage restorations and implant support crowns with different parameters accordingly this study fills the gap regarding the two parameters plus new materials that are supposed to be final restorations.(6,13,18)

This is a vitro experimental study with some limitations. The using of a stereomicroscope in measurement the marginal gap has difficult to pinpoint exact measurement points, distinguish between the tooth and cement, and identify the deepest part of the crown margin (12).

Furthermore, fracture resistance is merely a static loading test, whereas the clinical performance of dental crowns involves more complex, multidirectional forces. It is recommended that further research should be commenced to investigate these resin materials in clinical trial study design. Moreover, further research should be undertaken to investigate different physical and mechanical properties of all additively manufactured resins in the market.

CONCLUSIONS

1- VITA Enamic and Varseosmile demonstrated statistically significant greater resistance to fracture compared to Flexcera.
2- Additively manufactured materials (Flexcera and VarseoSmile plus) exhibited the least marginal gap as compared to the milled VITA Enamic.
3- All resin-based materials tested in this study showed a statistically significant increase in marginal gaps after thermomechanical aging.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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