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Evaluation of Microhardness of 3D Printable Permanent Dental Restorative Materials

Üç Boyutlu Yazıcılarda Kullanılan Daimi Dental Restoratif Materyallerin Mikrosertlik Açısından Değerlendirilmesi

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ABSTRACT

Objectives: This study aimed to evaluate the microhardness of 3D-printed permanent restorative materials exposed to commonly consumed beverages.

Materials and Method: Three different 3D-printed permanent restorative materials (Crowntec, SAREMCO; Permanent Crown Resin, FORMLABS; VarseoSmile TriniQ, BEGO) were used to prepare 120 disk-shaped (10×2mm) specimens and one CAD/CAM block (Cerasmart, GC) was used to prepare 40 specimens (n=10). Designs were created with SolidWorks 2023, and 2mm slices were obtained from the GC block (12x14x18mm). Specimen surfaces were polished with aluminum oxide disks (Sof-lex, 3M) and a diamond-impregnated polishing system (Eve Diacomp-Plus). Specimens were subjected to thermocycle (5-55°C, 5000 cycles) in distilled water, according to ISO/TS 11405:39, simulating six months of clinical aging. They were then immersed in distilled water, coffee, cola, and cherry juice corresponding to six months. Microhardness (VHN) was measured at baseline (t0), after thermal cycling (t1), and after immersion (t2) using a Vickers tester. Data were analyzed by three-way ANOVA and Tukey HSD post hoc test ($p<0.05$).

Results: Cerasmart showed the highest and VarseoSmile TriniQ the lowest VHN values among all groups and times. Permanent Crown Resin had significantly higher VHN in coffee and distilled water than in cola and cherry juice ($p<0.01$). Solution had no effect on VHN for VarseoSmile TriniQ, Crowntec, or Cerasmart ($p>0.05$).

Conclusion: Within the limitations of this study, the microhardness values of the tested 3D permanent restorative materials were affected by coffee, cola, and cherry juice, leading to a decrease in microhardness.

Keywords: *Beverages, Dental materials, Hardness, Three-dimensional printing*

ÖZET

Amaç: Bu çalışmanın amacı, üç boyutlu yazıcılarla üretilen daimi restoratif materyallerin günlük hayatta sıkça tüketilen içeceklere maruziyetinin mikrosertlik değerlerine etkisinin değerlendirilmesidir.

Gereç ve Yöntem: Üç farklı 3-B daimi restoratif materyal (Crowntec, SAREMCO; Permanent Crown Resin, FORMLABS; VarseoSmile TriniQ, BEGO) kullanılarak 10x2 mm disk şeklinde 120 adet örnek ve CAD/CAM blok (Cerasmart, GC) kullanılarak 40 adet örnek (n=10) hazırlandı. Üç boyutlu yazıcıda üretilen materyallerin dijital tasarımları SolidWorks 2023 programı ile yapıldı. GC bloktan (12x14x18mm) 2mm kalınlığında kesitler alındı. Örneklerin yüzeyleri alüminyum oksit kaplı diskler (Sof-lex, 3M) ve elmas emdirilmiş çift aşamalı polisaj sistemi (Eve Diacomp-Plus) kullanılarak cilalandı. Örnekler, ISO/TS 1140539 standartlarına göre 5/55°C distile suda 5000 devirde (6 aylık yaşlandırmaya denk gelen) termal döngüye maruz bırakıldıktan sonra distile su, kahve, kola ve vişne suyunda 6 aylık klinik kullanıma denk gelen sürede bekletildi. Mikrosertlik (VHN) değerleri başlangıç (t0), termal döngü sonrası (t1) ve solüsyonlarda bekletme sonrası (t2) olmak üzere 3 farklı zamanda ölçüldü. Veriler üç yönlü varyans analizi (ANOVA) ve Tukey HSD post hoc testi ile değerlendirildi ($p<0,05$).

Bulgular: Tüm solüsyon gruplarında ve tüm ölçüm zamanlarında en yüksek VHN değerleri Cerasmart örneklerde görülürken, VarseoSmile TriniQ en düşük değerleri sergiledi. Permanent Crown Resin kahve ve distile suda, kola ve vişne suyuna göre anlamlı derecede yüksek mikrosertlik değerleri sergiledi ($p<0,01$). VarseoSmile TriniQ, Crowntec ve Cerasmart için farklı solüsyonların mikrosertlik üzerine anlamlı bir etkisi gözlenmedi ($p>0,05$).

Sonuç: Bu çalışmanın limitasyonları dahilinde, kullanılan 3-B daimi restoratif materyallerin mikrosertlik değerleri kahve, kola ve vişne suyundan etkilendi ve mikrosertlik değerlerinde azalma görüldü.

Anahtar Kelimeler: *Dental materyal, İçecek, Sertlik, Üç boyutlu yazıcı*

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Introduction

In recent years, three-dimensional (3D) printing technology, integrated into dentistry, has introduced a new approach to the fabrication of permanent dental restorations. Compared to traditional methods, this technology offers more precise, faster, and reproducible results.[1,2]

3D printers build materials layer by layer using computer-aided design (CAD) data, facilitating the production of complex geometries.[3] In dentistry, 3D printing technology provides advantages such as reduced material waste, shorter production times, and cost-effectiveness.[4,5] The mechanical properties of dental restorative materials produced by this technology play a critical role in their clinical success.[6]

The mechanical behavior of 3D printed dental materials are highly sensitive to a variety of production parameters. Among the most critical are layer thickness, post-curing time, washing protocol, and resin processing conditions during fabrication.[7] Layer thickness directly impacts polymerization depth, interlayer adhesion, and dimensional stability.8 Post-curing facilitates further polymerization of residual monomers, reducing internal stresses and increasing cross-linking density, thereby improving the final strength and biocompatibility of the material.[9]

Microhardness is an important parameter indicating the wear resistance and surface durability of dental materials.[6] Materials with low microhardness are more susceptible to surface damage during toothbrushing or mastication of hard foods.[4,10] Therefore, evaluating the microhardness properties of permanent dental restorative materials used in 3D printers is of great clinical significance.

Chemical and thermal changes in the oral environment can affect the microhardness values of dental materials.[11,2] Exposure to different beverages may alter the surface properties and mechanical strength of dental restorative materials.[12] In particular, acidic beverages can soften the resin matrix and decrease microhardness.[13,14]

There is limited information regarding the mechanical properties of 3D-printed permanent dental materials.[3] The effects of different solutions on these materials are not yet fully understood.[15] Studies investigating the microhardness changes of dental restorative materials fabricated by 3D printers in different environments are scarce, and existing research reports conflicting results.[11] To better predict the clinical performance of these materials, it is essential to evaluate their microhardness properties after exposure to various beverage solutions.

The aim of this study was to evaluate the microhardness properties of three different 3D-printed permanent dental restorative materials after immersion in different beverage solutions (cola, coffee, cherry juice, and distilled water).

The null hypotheses of the study were as follows:

1. Beverage type does not affect the microhardness value of 3D printed permanent restorative material.
2. Type of 3D printed permanent restorative material does not affect the microhardness value in different solutions.

Material and Method

Ethical approval was not required for this study as it did not involve any interventions on human participants, the use of personal data, or animal experiments.

Disc-shaped specimens (10x2mm) were designed (CAD Software, Solidworks) and 3D printable resins (Crowntec, SAREMCO; Permanent Crown Resin, FORMLABS; VarseoSmile TriniQ, Bego) (Table 1) were produced using 3D printers (Asiga Max2, Formlabs Form 3D+,Varseo XS 3D, respectively) to be immersed in cola (CC), coffee (CO), cherry juice (CJ), and distilled water (DW) (Table 2) (n=10). Additionally, 40 specimens were obtained by sectioning hybrid ceramic blocks (Cerasmart, GC) into 2x12x14mm slices, resulting in a total of 160 specimens (N=160). Power analysis was performed using the GPower v3.1.9.7 program with a 95% confidence level (1-α), 95% test power (1-β), and an effect size of f = 0.40, a minimum of 10 samples per group (16) should be included, totaling a minimum of 160 samples in the study (Figure 1).

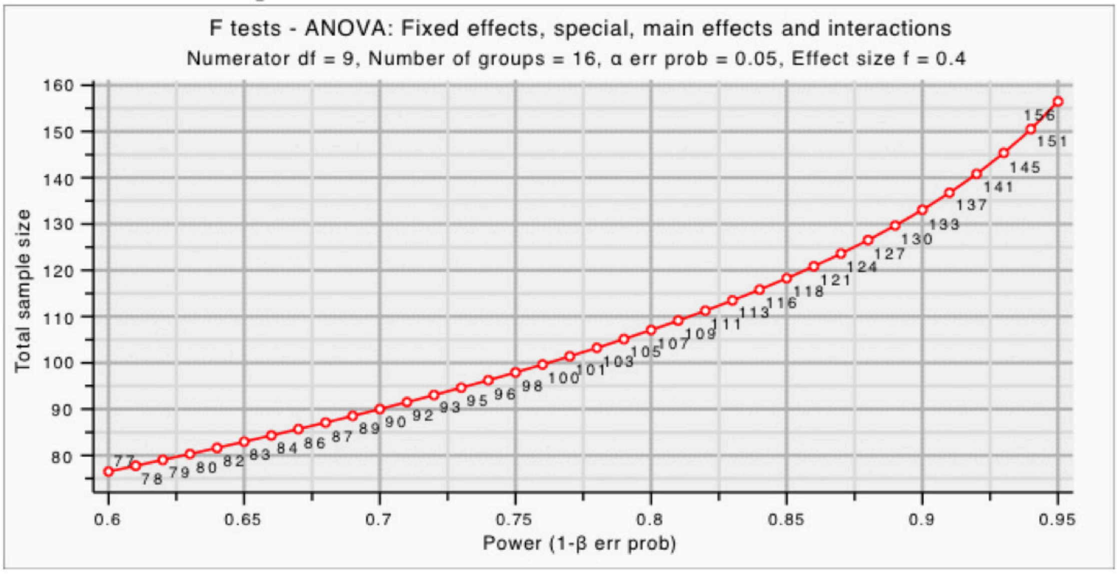

Figure 1. Power Analysis using the v3.1.9.7 GPower program.

All specimen surfaces were polished with aluminum oxide-coated disks (Sof-lex, 3M) and a diamond-impregnated two-step polishing system (Eve Diacomp-Plus). Initial microhardness measurements were performed using a Vickers microhardness tester (INNOVATEST 412A, Holland) with a load of 1000 g applied for 15 seconds, and baseline (t0) values were recorded (VHN).

Table 1. Characteristics of the tested restorative materials.

Material	Color	Manufacturer	Type	Composition	Polymerization Type	Production Method
Crowntec	A2	SAREMCO, Switzerland	3D-printed resin	UDMA-based resin composite	Light-cured + Post-curing	3D Printing (Max2, Asiga)
Permanent Crown Resin	A2	FORMLABS, USA	3D-printed resin	Methacrylate-based resin composite	Light-cured + Post-curing	3D Printing (Form 3D+, Formlabs)
VarseoSmile TriniQ	A2	BEGO, Germany	3D-printed resin	Ceramic-filled resin composite	Light-cured + Post-curing	3D Printing (Varseo XS 3D, BEGO)
Cerasmart	A2	GC, Japan	Hybrid ceramic CAD/CAM block	Flexible nano-ceramic matrix (resin + ceramic)	Pre-polymerized (industrial)	Milling (MCX5 Unit, Dentsply Sirona)

Abbrevations: 3D=Three dimensional, UDMA= Urethane Dimethacrylate

Specimens were then subjected to 5000 thermal cycles between 5°C and 55°C in distilled water, simulating six months of clinical aging according to ISO/TS 11405:39 standards. Following thermal cycling (t1), microhardness was remeasured. Subsequently, specimens were immersed in distilled water, coffee, cola, or cherry juice at 37°C for six days corresponding to six months of clinical exposure and final microhardness values (t2) were obtained.

Table 2. Group abbreviations, company details, and ph values of the tested beverages.

Beverage		Company	pH
Distilled Water	DW	N/A	7.0
Coffee	CO	Nestle, Switzerland	5.0
Cola	CC	The Coca Cola Company, USA	2.5
Cherry Juice	CJ	Dimes, Türkiye	3

Statistical analysis was conducted using SPSS version 26.0 (IBM SPSS, Türkiye). Three-way analysis of variance (ANOVA) was employed to identify significant differences in microhardness values according to material, solution and time. Post hoc comparisons were performed using the Tukey HSD test. Statistical significance was set at $p<0.05$.

Results

The effects of material type, solution type, and their interaction on microhardness values are summarized in Table 3. Statistically significant main effect of material type ($p<0.001$) and solution type ($p=0.002$) was observed on microhardness values, while the material × solution interaction was not significant ($p=0.252$).

Table 3. Effect sizes for material type, solution type, and their interaction on microhardness values

	F	p
Material	20332.562	<0.001
Solution	5.016	0.002
Time	36.815	<0.001
Material × Solution	1.281	0.252
Material × Time	8.365	<0.001
Solution × Time	1.040	0.399
Material × Solution × Time	1.976	0.011

*Three-way ANOVA

The microhardness values of the materials obtained according to the solutions at different times are presented in Table 4. At baseline (t0), the highest mean VHN value was observed for Cerasmart (75.87), followed by Permanent Crown Resin (25.62), Crowntec (25.04), and VarseoSmile TriniQ (18.51).

After thermocycle (t1), Cerasmart maintained the highest microhardness value (75.82), followed by Permanent Crown Resin (24.98), Crowntec (24.58), and VarseoSmile TriniQ (18.34). While a slight decrease was observed in the microhardness values of all materials compared to baseline, the overall ranking remained consistent. At the final time point (t2), Cerasmart again exhibited the highest microhardness value (75.96), followed by Permanent Crown Resin (25.11), Crowntec (24.80), and VarseoSmile TriniQ (18.52).

Following immersion period (t2), significant differences in microhardness values were observed among different solutions ($p < 0.05$). Samples stored in distilled water and cola exhibited the highest overall mean microhardness values (36.39 and 36.44, respectively), with no statistically significant difference between them (p >0 .05). These groups were followed by cherry juice (35.81) and coffee (34.55), which formed a statistically similar subgroup. The lowest mean VHN value was recorded in specimens stored in CJ, specifically for VarseoSmile TriniQ (18.48).

Table 4. The microhardness values of the materials obtained according to the solutions at different times.

Time	Material	CC^{A}	CJ	CO^{B}	DW^{B}	Total Mean±SD
T_0	VarseoSmile TriniQ ^{a}	18.41 ± 0.25	18.51 ± 0.13	18.61 ± 0.20	18.50 ± 0.19	18.51 ± 0.20
	Permanent Crown Resin ^{b}	24.82 ± 0.79	25.10 ± 0.75	26.56 ± 0.79	26.00 ± 0.94	25.62 ± 1.06
	Crowntec ^{b}	25.25 ± 1.40	24.58 ± 1.34	25.16 ± 1.48	25.16 ± 1.50	25.04 ± 1.40
	Cerasmart^{c}	74.64 ± 2.63	75.52 ± 1.98	76.41 ± 1.30	76.93 ± 1.72	75.87 ± 2.09
	Total Mean±SD	35.78 ± 22.93	35.92 ± 23.33	36.69 ± 23.45	36.65 ± 23.77	36.26 ± 23.15
T_1	VarseoSmile TriniQ ^{a}	18.26 ± 0.24	18.34 ± 0.11	18.42 ± 0.11	18.36 ± 0.13	18.34 ± 0.16
	Permanent Crown Resin ^{b}	24.14 ± 0.73	24.64 ± 0.66	25.70 ± 0.48	25.42 ± 0.80	24.98 ± 0.91
	Crowntec ^{b}	24.69 ± 1.53	24.20 ± 1.45	24.82 ± 1.39	24.62 ± 1.26	24.58 ± 1.38
	Cerasmart^{c}	74.96 ± 2.49	75.39 ± 1.74	76.28 ± 1.13	76.67 ± 1.69	75.82 ± 1.89
	Total Mean±SD	35.51 ± 23.25	35.64 ± 23.41	36.30 ± 23.56	36.27 ± 23.81	35.93 ± 23.29
T_3	VarseoSmile TriniQ ^{a}	18.51 ± 0.31	18.48 ± 0.28	18.52 ± 0.14	18.57 ± 0.11	18.52 ± 0.22
	Permanent Crown Resin ^{b}	24.56 ± 0.93	24.62 ± 0.68	25.66 ± 0.56	25.62 ± 0.83	25.11 ± 0.91
	Crowntec ^{b}	24.83 ± 1.49	24.37 ± 1.25	25.09 ± 1.41	24.91 ± 1.11	24.80 ± 1.30
	Cerasmart^{c}	75.11 ± 2.13	75.77 ± 1.83	76.52 ± 0.95	76.47 ± 1.72	75.96 ± 1.75
	Total Mean±SD	35.75 ± 23.19	35.81 ± 23.52	36.44 ± 23.62	36.39 ± 23.62	36.10 ± 23.27

Different superscript letters (lowercase: a, b, c for materials; uppercase: A, B for solutions) indicate statistically significant differences ($p<0.05$). No significant differences were found between groups sharing the same superscript letter.

Discussion

The first null hypothesis stated that there would be no statistically significant difference in microhardness among the different materials tested. However, the ANOVA results revealed a highly significant effect of material type on microhardness ($p<0.001$), with Cerasmart exhibiting the highest values and VarseoSmile TriniQ showing the lowest. Therefore, H□ was rejected, indicating that the microhardness properties of the materials differed significantly according to their chemical composition and production method.

The superior microhardness values of Cerasmart CAD/CAM block can be attributed to the higher degree of polymerization and lower residual monomer content typical of CAD/CAM-manufactured materials.[16] Goujat[17] evaluated the mechanical properties of four CAD-CAM block materials, including Cerasmart and IPS e.max CAD. Their findings indicated that Cerasmart exhibited superior flexural strength compared to other tested materials. These results align with this study, where Cerasmart demonstrated the highest microhardness values and maintained its mechanical integrity after thermal aging. The consistency between these studies reinforces the clinical reliability of Cerasmart as a durable restorative material.

The low microhardness values of the VarseoSmile TriniQ may be associated with incomplete polymerization and the presence of residual monomers in 3D-printed resin materials.[4,6,18]

Barutcugil[19] investigated the potential monomer release from resin-based CAD/CAM composite blocks and highlighted that insufficient polymerization or lower cross-linking density can lead to detectable monomer elution. VarseoSmile TriniQ exhibited consistently lower microhardness values compared to Cerasmart, potentially indicating a lower degree of polymer cross-linking and aligning with the findings. These results underline the importance of optimized post-curing protocols to minimize residual monomer content and enhance the mechanical stability of 3D-printed restorative materials.

Yilmaz Atali[20] reported that the degree of polymerization significantly influences the microhardness of single-shade universal resin composites and demonstrated a positive correlation between the degree of conversion and Vickers hardness values, emphasizing that insufficient polymerization results in lower surface hardness and potentially compromised mechanical performance. While their evaluation focused on conventionally light-cured composites, the observed trends align with the current results, indicating that both curing intensity and time are key factors in achieving optimal surface integrity. These findings support the notion that achieving an adequate degree of polymerization through post-curing is essential.

The similar microhardness values observed between Permanent Crown Resin and Crowntec suggest that these two materials may possess comparable chemical compositions and polymerization characteristics. Consistently, Demirsoy[21] reported that Crowntec showed similar microhardness values across different solutions.

The second null hypothesis proposed that there would be no statistically significant difference in microhardness among the different solutions. Although the ANOVA showed a statistically significant effect of solution type (p=0.002), Tukey HSD post hoc comparisons revealed that significant differences were observed only between certain pairs (specifically between coffee and cola). Thus, while H_2 was also rejected, the magnitude of the effect of the solutions on microhardness was relatively small compared to the material effect.

In this study, Permanent Crown Resin material specimens immersed in coffee and distilled water exhibited significantly higher microhardness values compared to those immersed in cola and cherry juice. This finding suggests that acidic beverages (cola and cherry juice) may soften the resin matrix, leading to decreased microhardness values. Santi[22] similarly reported that acidic environments can soften resin matrices and adversely affect mechanical properties. However, no significant effect of different solutions on microhardness was observed for the VarseoSmile TriniQ, Crowntec, and Cerasmart materials. This outcome indicates that the chemical structure of these materials may confer greater resistance to environmental degradation.[23]

Likewise, Demirsoy[21] found no significant differences in Vickers hardness values for Saremco Crowntec specimens immersed in wine and coffee solutions.

All specimens in this study were subjected to 5000 thermal cycles between 5°C and 55°C, following ISO/TS 11405:39 standards. A general decrease in microhardness values was observed after thermal cycling (t1), suggesting that thermal cycling can affect the surface properties of materials. Falahchai[7] similarly reported that thermal cycling impacted the mechanical properties of 3D-printed denture base materials. However, in the study by Yıldırım[24], an increase in average hardness values after thermal cycling was observed only in 3D-printed composite resin materials. This discrepancy may be attributed to differences in material compositions and variations in thermal cycling protocols.

Temizci[25] demonstrated that thermocycling had no significant effect on the mechanical properties of Crowntec, Permanent Crown Resin, and Cerasmart. Similarly in this study, these materials maintained their microhardness values although a slight decrease was observed after thermal cycling, with Cerasmart consistently exhibiting the highest hardness values. The consistency of outcomes across both studies supports the thermal and mechanical reliability of these materials and underscores their suitability for long-term clinical applications.

The results of this study suggest that microhardness should be considered when selecting materials for permanent dental restorations. Particular attention should be paid when using materials susceptible to acidic degradation, such as Permanent Crown Resin, especially in patients with high acidic beverage consumption. Materials like Cerasmart, which exhibit high microhardness values, may be preferable in clinical situations requiring superior wear resistance and surface durability.

Moreover, to optimize the clinical performance of 3D-printed permanent dental restorative materials, appropriate post-curing protocols and optimization of printing parameters are essential.

This study has several limitations. Only microhardness properties were evaluated, while other mechanical properties (such as flexural strength and fracture toughness) were not assessed. Additionally, the color stability of the 3D printed materials was not evaluated. Since post-curing parameters, material composition, and photo initiator systems can significantly influence the optical properties of resin-based materials, including discoloration over time, future studies should incorporate objective color measurements using spectrophotometric methods such as the CIEDE2000 system to better assess the long-term esthetic behavior of 3D printed resins. Third, the immersion period in different solutions was limited, and long-term effects were not evaluated. Long-term clinical studies are necessary to evaluate the mechanical and physical properties of 3D-printed permanent dental restorative materials.

Conclusion

The findings of this study revealed that the microhardness values of the tested 3D-printed permanent dental restorative materials vary depending on the material type, and certain materials may be affected by acidic beverages. Cerasmart demonstrated the highest microhardness values, whereas VarseoSmile TriniQ the lowest. Solutions also had a significant but smaller effect on microhardness, with coffee causing less degradation compared to cola. These results indicate that microhardness properties should be considered when selecting materials for permanent dental restorations.

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Ethical Approval

Ethical approval was not required for this study as it did not involve any interventions on human participants, the use of personal data, or animal experiments. The study was conducted in accordance with relevant regulations and ethical principles.

Conflict of Interest

The authors declare no conflicts of interest regarding the subject matter or materials discussed in this article.

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Authorship Contributions

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