



THE EVALUATION OF THE SURFACE MICRO-HARDNESS VALUES IN DIFFERENT DEPTH OF RESIN BLOCKS

Rezin Blokların Farklı Kesitlerindeki Yüzey Mikrosertlik Değerlerinin Değerlendirilmesi

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ABSTRACT

Objective: The aim of this study was to investigate the polymerization depth and hardness of each 2 mm layer of resin blocks.

Materials and Methods: Two composite resin block materials (Cerasmart, GC Corp., Tokyo, Japan and Lava Ultimate, 3M ESPE, St. Paul, MN, USA) and one hybrid resin block material (Vita Enamic, Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany) were included in the present study. Using a diamond saw (Exakt 300 cl Apparatebau, Norderstedt, Germany), 10×10×2 mm blocks were prepared from the upper surface to the lower surface, under water-cooling. All specimens were abraded by using 500,1200,2500 grid SiC abrasive papers (Exakt 400 cs Apparatebau, Norderstedt, Germany), under water-cooling. After storage in distilled water at 37 °C for 24 hours, Knoop hardness test was performed using a hardness testing machine (Buehler MMT 3 digital micro hardness tester, Lake Bluff, IL, USA) (500 gf, 10s).

Results: There was a statistically significant difference between the materials in terms of surface hardness ($p<0.05$). The highest hardness value was obtained on Vita Enamic block, whereas the lowest group was the Cerasmart. The surface hardness values were Vita Enamic> Lava Ultimate >Cerasmart, respectively. No difference was found among slices of 2 mm thickened specimens of each material's blocks ($p>0.05$)

Conclusion: The hardness measurements of the layers of blocks were similar.

Key Words: CAD/CAM, composite resin blocks, microhardnes

ÖZ

Amaç: Bu çalışmanın amacı rezin blokların 2 mm'lik her katmanındaki polimerizasyon derinliklerinin ve sertliklerinin araştırılmasıdır.

Gereç ve Yöntemler: Mevcut çalışmada iki farklı marka kompozit rezin blok (Cerasmart, GC Corp., Tokyo, Japonya ve Lava Ultimate, 3M ESPE, St. Paul, MN, ABD) ve bir hibrit rezin blok (Vita Enamic, Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Almanya) kullanıldı (n=5). Bir elmas testere ile su soğutması altında (Exakt 300 cl Apparatebau, Norderstedt, Almanya) blokların üst yüzeyinden alt yüzeyine doğru 10×10×2 boyutlarında horizontal kesitler alındı (Exakt 300 cl Apparatebau, Norderstedt, Almanya). Örnekler su altında sırasıyla 1000, 1500, 2500 grit zımpara kullanılarak polisajlandı (Exakt 400 cs Apparatebau, Norderstedt, Almanya). Örneklerin Knoop yüzey sertlik ölçümü 37° C'lik distile suda 24 saat süreyle bekletildikten sonra bir mikrosertlik test cihazı (Buehler MMT 3, Lake Bluff, IL, ABD) kullanılarak yapıldı (500 gf, 10s).

Bulgular: Materyallerin sertlik ölçümleri arasında istatistiksel olarak anlamlı bir fark vardır ($p<0,05$) En yüksek sertlik değeri Vita Enamic blok, en düşük Cerasmart materyalinde ölçülmüştür. Yüzey sertliğinin derecesi sırasıyla Vita Enamic> Lava Ultimate >Cerasmart şeklindedir. Bloklarının 2 mm kalınlığındaki kesitleri arasında fark bulunmadı ($p> 0,05$).

Sonuç: Blok katmanlarının sertlik ölçümleri benzerdir. Blokların tüm katmanları, oklüzal kuvvetlere karşı koyacak kadar serttir.

Anahtar Kelimeler: CAD/CAM, kompozit rezin blok, mikrosertlik

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INTRODUCTION

Dental materials are widely used in all areas of routine dental practice. There are mainly two methods for their application. Firstly, dental biomaterials are placed into living tissues, such as teeth, to fill the space. Furthermore, inlay, onlay, crown and bridge restorations are manufactured using a variety of materials to recover the morphology and functioning of the teeth.

These materials can be dissolved by the saliva and they may also have cytotoxic potential due to residual monomers after curing.

During the last decade, a remarkable increase has been seen in the use of computer-aided design and manufacturing (CAD/CAM) in dentistry. For this purpose, various methods have been developed to obtain ceramic and polymer or composite resin hybrid materials.

These commercial dental CAD/CAM blocks have been frequently used to utilize single step restorations in dentistry. Two main types of esthetically pleasing CAD/CAM processed dental materials were used for indirect dental restorative purposes; glass-ceramic/ceramics and resin composites.

Although glass-ceramics/ceramics blocks have some advantages such as aesthetic appearance (attractiveness), biocompatibility, low fracture toughness, high resistance to failure against different chemicals, but have some disadvantages such as high stiffness and low strength to chewing force.^{1,2,3}

Single tooth restorations produced using ceramic materials showed an overall failure rate of approximately 22% after 4 years in service.⁴ Dense ceramics are characterized by high hardness and wear resistance values; however, they cannot withstand elastic deformation because of their Young modulus, which is higher than dental tissues.⁵ According to a recent studies using this ceramic materials indicated a flexural strength of greater than

100 MPa, obtained with a three point bending test.^{6,7} The obtained flexural strength value is higher than the critical value proposed by International Organization for Standardization (ISO) 6872 for single unit restorations.⁸

Resin blocks have a higher modulus of resilience than ceramic blocks therefore they cause less wear on the opposing teeth.^{9,10} Moreover, due to the young modulus of resin blocks are closer to dentin, the risk of fracture and chipping is lower.^{1,9-12} Additionally, they are easier to repair and polish than glass-ceramics.¹³⁻¹⁷ Industrial production of these blocks under high temperature and high pressure has led to a higher conversion degrees (85%) and higher volume fraction filler.^{5,18,19}

These high-density polymer resin blocks are polymerized under controlled and standardized industrial conditions with high pressure and high temperature. The production and structure of resin blocks are different and they are polymerized under different temperature and pressure condition.²⁰ Vita Enamic is composed of a porous ceramic network (86%), which is then infiltrated with a polymer by capillary action. Hybrid blocks containing nanoceramic particles bound in the polymeric matrix either nanofillers (Lava Ultimate; 3M ESPE, St. Paul, MN, USA) or nanohybrid fillers (Cerasmart; GC Corp., Tokyo, Japan).

The polymerization of resin composite material under HP/HT (High Pressure/High Temperature) resulted with superior properties in comparison with those of their photopolymerized counterparts in vivo conditions. Curing depth of composite resins intraoral are limited, no greater than 2 mm thick should be placed to ensure complete polymerization. the depth of cure of the composite resins affect the composition of residual monomers and their surface hardness.²¹

In most CAD / CAM systems restorations can be produced from prefabricated blocks by milling using bur, only diamond or diamond

discs. In this technique, 90% of the blocks are removed to achieve the desired restoration scheme. Different depth of layers of the resin blocks are used during to produce a restoration.

In this study the null hypothesis was there would be no significant differences in the surface hardness of different depth of various CAD/CAM composite resin blocks fabricated under HP/HT conditions.

MATERIALS AND METHODS

Two composite resin block materials and one hybrid resin block material were included in this study. The CAD/CAM block materials evaluated in this study were Cerasmart (CER; GC Corp., Tokyo, Japan; A3 LT/14), Lava Ultimate (ULT; CAD-CAM restorative; 3M ESPE, St. Paul, MN, USA; A3-HT/14L) and Vita Enamic (VIT; Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany; A3C/I14) (Table 1).

Table 1: Materials tested and their compositions.

Type	Brand	Code	Manufacturer	Shade/Si ze	Monomer	Filler Composition	Fillerwt (%)
Composite resin block	Cerasmart	CER	GC Corp., Tokyo, Japan	A3 LT/14	Bis-MEPP, UDMA, DMA	Silica (20 nm), barium glass (300 nm)	71
Composite resin block	Lava Ultimate	ULT	3M ESPE, St. Paul, MN, USA	A3-HT/14L	Bis-GMA, UDMA, Bis-EMA, TEGDMA	SiO2 (20 nm), aggregated SiO2-Al2O3-ZrO2 (200-600 nm) cluster (1-6 µm)	80
Hybrid ceramic block	Vita Enamic	VIT	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	A3C/I14	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide	86

Bis-GMA: Bisphenol A diglycidylether methacrylate; UDMA: Urethane dimethacrylate; Bis-EMA: Bisphenol A polyethylene glycol diether dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; Bis-MEPP: 2,2-Bis (4-methacryloxyphenyl) propane;

Square specimens (n=5/material) of approximate dimensions 10×10×2 mm blocks were sectioned (5 slices/block) from commercially available CAD-CAM materials using a diamond saw (Exakt 300 cl Apparatebau, Norderstedt, Germany) with water cooling. All specimens were abraded followed by 500, 1200, 2500 grid using SiC abrasive paper (Exakt 400 cs Apparatebau, Norderstedt, Germany). After storage in distilled water at 37 °C for 24 hours the Knoop hardness test was performed using a hardness testing machine (Buehler MMT 3 digital micro hardness tester, Lake Bluff, IL, USA) using a

load of 500 gf and a loading time of 10s. The hardness of different depth of 2 mm thickened slices of evaluated materials was measured for each of 5 repeated blocks and nine indentations were applied in specific locations for each slice (Figure 1).

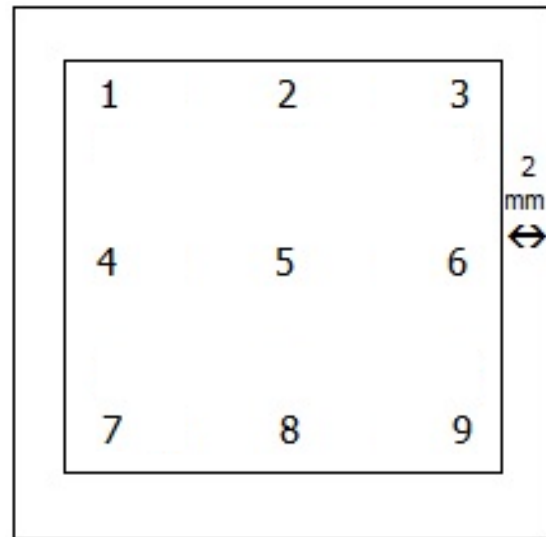


Figure 1: Indentations for each slice

Statistical analyses

Statistical analyses were performed using SPSS v. 21.0 (IBM Corp., Armonk, NY, USA). The normality assumption for micro-hardness data tested by using Shapiro-Wilk test, the assumption was not confirmed. Therefore, the non-parametric Kolmogorov-Smirnov test was conducted to evaluate micro hardness of materials and also different slices of blocks. Adjusted pairwise comparisons were further performed to evaluate inter-group differences among groups with value of statistical significance α=0.05. The mean values, standard deviations, medians, and minimum, maximum values of materials and five slices of each material were given as descriptive statistics.

RESULTS

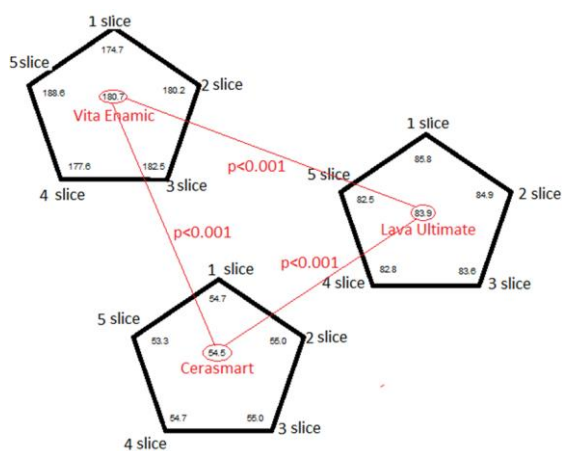
The highest hardness value was obtained from Vita Enamic block material, while the lowest was obtained from the Cerasmart. The obtained values were shown in Table 2.

Table 2: Descriptive Statistics for hardness

CAD/CAM Material	N	Mean	Std. Deviation	Median*	Min	Max
Cerasmart	225	54.5	4.1	54.1 ^a	43.7	71.6
Vita Enamic	225	180.7	29.1	176.0 ^b	122.7	295.7
Lava Ultimate	225	83.9	6.6	83.2 ^c	69.5	113.2

*Identical superscript letters indicate that are not significantly different (Adj. Sig: $p > 0.05$)

The post hoc test revealed a significant difference among all the tested materials ($p < 0.05$) with the following ranking: Vita Enamic > Lava Ultimate > Cerasmart. No difference was found among slices of 2 mm thickened specimens of each material's blocks ($p > 0.05$) (Graph 1).



Graph 1: Clustering groups and descriptive Statistics of the hardness of different depth of 2 mm thickened slices of Vita Enamic, Lava Ultimate and Cerasmart

DISCUSSION

The surface hardness testing is useful for evaluating the properties of composites in relation to the depth of polymerization.^{22,23,24}

Brinell, Knoop, Barcol, Rockwell and Vickers are the most commonly used test methods for surface hardness measurements of composite resin based restorative materials used in dentistry. The choice of the hardness test to be applied depends on the materials and the hardness value expected from the material.

Knoop hardness test is the most commonly used method for evaluating polymeric materials, such as resin composites, because it uses a rhombic diamond tip and reduces the effect of elastic recovery after removal of the tip.²⁵ For this reason we have

decided to use the Knoop hardness test to determine the hardness of CAD/CAM composites.

Vita enamic has the highest hardness value due to the both filler amount and ceramic content. Lava ultimate and cerasmart contain similar components but the content of inorganic fillers is approximately 80% in Lava Ultimate and 71% in GC cerasmart.

The hardness is measured higher than cerasmart because of the filler content of the Lava ultimate. Hardness order is similar in other studies, conducted with the materials used in the present study.^{26,27,28} But it is unclear from which region the hardness test was conducted in these studies.

In the present study, although there is no statistical difference, hardness values are different in different layers of the same block- ($P > 0.05$). The null hypothesis is accepted. There isn't significant differences in the surface hardness of different depth of various CAD/CAM composite resin blocks. When the SEM images of the conducted studies are taken into account, the filler and ceramic distributions and shapes of the resin blocks are not homogenous, so the hardness results may vary in different regions of the blocks.^{25,28} These results shows that the hardness is not only affected by the inorganic filler content but also by the filler size, the filler form and the polymeric matrix.

Knoop hardness test method are used to evaluate the bright and smooth surface specimens. Therefore, the hardness measurements of the specimens were not performed on the top layer of the resin blocks because of their roughness in the present study.

In the Lava Ultimate groups, a cloud-like shadow formed around the rhombic diamond on the microscopic image.

It is estimated that this view is due to fracture of feldspar ceramic filler. It is thought that these ceramic fillers have increased

hardness but also increased in the possibility of fracture and chipping.

Lack of this study no thermocycling done in the samples. If the thermocycling was done, the results could be different from the samples with more water absorption.

CONCLUSION

The hardness measurements of the layers of blocks were similar. All layers of blocks are hard enough to resist defatation by occlusal forces.

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