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Evaluation of Correlation Between Wear Resistance and Microhardness of Resin-based CAD/CAM Blocks

Kompozit İçerikli Cad/Cam Blokların Aşınma Direnci ile Mikrosertlik Arasındaki Korelasyonun Değerlendirilmesi

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Abstract

Aim: Evaluating the relation between wear resistance and microhardness of composite blocks, hybrid block, and resin composite to provide convenient argument for clinical application.

Methods: A conventional resin composite (IPS Empress Direct, Ivoclar Vivadent, Liechtenstein), a hybrid ceramic block (Enamic, Vita, Germany), and composite blocks (Lava Ultimate, 3M, USA; Hc block, Shofu, Japan; Brilliant Crios, Coltene, Switzerland; Cerasmart, GC Corp., Japan) were investigated. Specimens (n=12 for each) were loaded in a chewing simulator (Dent Ar-Ge, Analitik Medical, Turkey) for thermal cycling (49 N force, 240.000 cycles, 1.5 mm lateral movement, 1.7 Hz frequency) and worn sufaces were scanned with Las-20 (Laser scanner, SD-Mechatronic, Germany), and Vickers microhardness (VHN) values were determined (200 grf/10 sec). Statistical analysis was performed using Spearman Correlation Coefficient, Kolmogorov-Smirnov, Mann-Whitney U, Friedman and Kruskal-Wallis tests ($p<0.05$).

Results: Significant correlation was detected between microhardness and wear resistance for all composite Cad/Cam blocks ($p<0.001$), whereas no correlation was observed for hybrid block and resin composite material ($p\geq0.05$). Vita Enamic showed the highest VHN value and wear resistance among all the materials ($p<0.001$).

Conclusion: Within the limitations of this *in vitro* study, a significant correlation between microhardness and wear resistance was observed only for composite Cad/Cam blocks which varied among the brands used in this study.
Keywords: Cad/Cam Block, Chewing simulator, Microhardness, Resin Composite, Wear

Öz

Amaç: Klinik uygulamalara rehberlik sağlamak amacıyla kompozit bloklar, hibrid blok ve rezin kompozit materyallerin aşınma direnci ve mikro sertlik arasındaki ilişkinin değerlendirilmesi.

Yöntem: Geleneksel rezin kompozit (IPS Empress Direct, Ivoclar Vivadent, Liechtenstein), hibrid seramik blok (Enamic, VITA, Almanya) ve kompozit bloklar (Lava Ultimate, 3M, ABD; Hc blok, Shofu, Japonya; Brilliant Crios, Coltene, İsviçre; Cerasmart, GC Corp., Japonya) araştırıldı. Örnekler (n=12) termal siklus özelliği olan çiğneme simülatöründe (Dent Ar-Ge, Analitik Medikal, Türkiye) yaşlandırıldı (49 N kuvvet, 240,000 siklus, 1,5 mm lateral hareket, 1,7 Hz frekans). Lazer tarayıcı kullanılarak aşınan yüzeyler tarandı (Las-20, SD Mechatronic, Almanya) ve Vickers mikrosertlik değerleri (VHN) ölçüldü (200 grf, 10s). İstatistiksel analiz spearman korelasyon katsayısı, Kolmogorov-Smirnov, Mann-Whitney U, Friedman ve Kruskal-Wallis testleri kullanılarak yapıldı ($p<0,05$).

Bulgular: Tüm kompozit blokları değerlendirildiğinde, aşınma direnci ve mikrosertlik arasında anlamlı korelasyon tespit edildi ($p<0.001$). Ancak hibrid blok ve rezin kompozit gruplarında aşınma direnci ve mikrosertlik arasında bir korelasyon bulunamadı ($p\geq0.05$). Tüm materyaller içerisinde Vita Enamic en yüksek VHN değeri ve aşınma direncini gösterdi ($p<0.001$).

Sonuç: Bu *in vitro* çalışmanın şartları altında, yalnızca kompozit Cad/Cam blokta aşınma direnci ve mikrosertlik arasında anlamlı korelasyon gözlemlenmiş olup korelasyon durumu çalışmada kullanılan materyallere göre değişiklik göstermiştir.
Anahtar kelimeler: Aşınma, Cad/Cam Blok, Çiğneme simülatörü, Mikrosertlik, Rezin Kompozit

Introduction

Prefabricated polymers provide higher mechanical properties compared to direct resin composites. Industrial conditions provide homogeneus internal structure owing to keep high temperature and pressure parameters (1).

CAD/CAM (Computer aided design/Computer aided manufacturing) has shown a major development in last years in clinical dentistry. Computer programs and milling

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devices have been improved. CAD/CAM technology lead to the improvement of materials with polymeric matrix and use of polycrystalline ceramics (2). Various blocks having different physical properties are currently avaliable in the market (3). Resin nano ceramic (RNC) and a polymer infiltrated ceramic (PIC) materials can be used as alternatives to ceramics (4). RNC block has a polymer matrix reinforced with ceramic fillers. PIC materials consist of porous ceramic matrix infiltrated with polymers (4).

Direct resin composite restorations are popular options for clinical applications in daily practice (5,6). Composite resins may provide aesthetic outlook in a minimally invasive way (7). In order to succeed in resin composite restorations; correct adhesive protocol, proper selection of the resin-based material, and effective technique are required (8). The structure as well as the thermal expansion coefficient of components vary among the composite materials (9). As far as composite materials are affected by intraoral temperature and fluids, thermocycling was accepted as the universal aging method (10).

Clinical performance of restorative materials are determined by evaluating the materials mechanical properties, such as flexural strength, fracture toughness, diametral tensile strength, compressive strength, wear resistance, and surface hardness (11).

Intraoral tribology describes wear as the loss volume of tooth or restorative material, which occurs in consequence of the interaction between two surfaces (12). Intraoral wear is based on four fundamental mechanisms: corrosive wear, fatigue wear, two-body wear, and three-body wear. Two-body wear and three-body wear mechanisms were accepted as the basic mechanisms for composite restorative materials (13). Two-body wear mechanism was defined as the material loss when the surfaces are in contact without the existence of another object, while three-body wear was characterized with presence of a third body between two antagonistic surfaces (14). Clinical tests are necessary to define complicated oral wear circumstances, however these tests are time consuming and expensive and they don't permit examination of variable factors such as oral conditions or masticatory forces (15). Consequently *in vitro* chewing simulation has still been defined as a convenient resolution for evaluating the wear performance of dental restorative materials (16).

Microhardness test is one of the most common methods used for evaluating the surface hardness of resin composites due to the ease of application steps. There are four methods for microhardness evaluations: Brinell, Knoop, Rockwell and Vickers (17). Vickers hardness (VHN) test is the most frequently used method for resin composites among others. VHN method works by indenting the specimen with a diamond indenter, in the form of a pyramid with a square base of which opposing faces have a 136° angle. It has a test force between 1 gf to 100 kgf and the load is applied for 10-15 sec (18). The VHN number should be presented together with the dwell time and test force. Surface hardness is described as the resistance of the specimen to indentation. Surface hardness measurement is one of the markers of the degree of conversion, and as a result indicates the clinical performance of restorative materials (19).

Strength and rigidity of resin-based materials were considered to be influenced by surface characteristics. Surface hardness is one of the criteria to assess the wear resistance of the materials (20). The amount of wear was also reported to be related to the surface microhardness (21). Some researchers have found no correlation between hardness and wear resistance (18), whereas several researches have reported the presence of correlation between these mechanical properties (22,23).

The objective of this *in vitro* research was to determine the relation between abrasive wear resistance and surface microhardness of a resin-based composite (IPS Empress Direct, Ivoclar Vivadent, Liechtenstein), a hybrid ceramic block (Enamic, VITA, Germany), and three composite blocks (Brilliant Crios, Coltene, Switzerland; Hc Block, Shofu, Japan; Cerasmart, GC Corp., Japan; Lava Ultimate, 3M, US). The null (h_0) hypothesis stated that, there will be no correlation between microhardness and wear resistance of the materials tested.

Material and Method

Preparation of the specimens

IPS Empress Direct, Enamic, Brilliant Crios, Hc block, Cerasmart and Lava Ultimate groups (n=12 for each group) were investigated (Table 1). Resin composite specimens were placed in silicone molds under light cured (Valo, Ultradent Products, Switzerland) for 20 sec under finger pressure using mylar strips. The specimens were polished under running water using aluminum oxide (Al_2O_3) embedded discs (Sof-Lex, 3M, US) and the polishing speed was set at approximately 20.000 rpm. Coarse (100 µm abrasive particles), medium (40 µm abrasive particles), fine (24 µm abrasive particles), and super-fine (8 µm abrasive particles) discs were used, respectively.

The polishing process was performed by a single experienced operator following the manufacturer's instructions (under slight hand pressure, 20 seconds application per disc). For each specimen a new polishing disc was used. Composite and hybrid block specimens were cut into 3 mm thick slices using Isomed (Buehler Ltd., USA). For polishing, Minitech 233 (Presi, Grenoble, France) was used under running water (170 rev/min, 15 s).

Table 1. Materials tested in the study.

		Manufacturer	Monomer	Filler	
				Content	Mass (Volume %)
Composite block	HC Block	Shofu, Japan	.TEGDMA, UDMA	Zirconium silicate Silica-powder, micro fumed silica	61
	Cerasmart	GC, Japan	UDMA, Bis-MEEP, DMA	Barium glass and silica nanoparticles	71
	Lava Ultimate	3M, USA	UDMA, Bis-EMA TEGDMA, Bis-GMA,	Silica/zirconia nanoparticles	80
	Brilliant Crios	Coltene Switzerland	Bis-GMA, TEGDMA, BIS-EMA	Silica particles, barium glass	71
Hybrid block	Vita Enamic	Vita Zahnfabrik, Germany	UDMA, TEGDMA	PIC with feldspatic porcelain network material	86
Resin Composite	IPS Empress Direct	Ivoclar Vivadent, Liechtenstein	Dimethacrylate	Barium glass filler ytterbium trifluoride barium alumina	81,2

Aging procedure of the specimens

All specimens were embedded in holders using acrylic resin (Imicryl, Turkey). Using a chewing simulator (Dent Ar-Ge, Analitik Medical, Turkey) specimens were thermo-mechanically aged with a total of 240.000 chewing cycles against a stainless steel ball with a diameter of 3 mm. The chewing simulator was used in the present study had a thermal cycling feature. The force parameters were set at 49 N force, 1.5 mm lateral movement, 1.7 Hz frequency, and the thermocycle parameters were 5-55^{o}C hot / cold bath water temperature, 60 sec waiting time and approximately 1800 cycles.

Assessment of wear and microharness

Three dimensional surface analysis of worn surfaces of the specimens was captured using Las-20 (Laserscanner, SD Mechatronic, Germany) and the volumetric loss (µm) was calculated with three-point alignment method using a specific 3D processing software (Geomagic Control, 3D Systems Inc., Rock Hill, USA).

Following the thermomechanical aging, Vickers microhardness (VHN) was measured by using a microhardness tester (Wilson Wolpert Micro-Vickers 401MVD, Wilson Wolpert Instruments, Germany) with the parameters of 200 gram force (grf) and 10 sec dwell time. Three measurements from different areas were made on the surfaces of the specimens and their average was recorded as the final VHN value. The VHN was determined by evaluating the length of the indentations and using the specific formula: H=1.854 P/d2 (p: load, d: diagonal length).

Statistical Analysis

Normality of input dispersion was evaluated using Kolmogorov-Smirnov test. The evaluation of wear was performed with Mann-Whitney U and Kruskal-Wallis tests. Evaluation of microhardness was determined with Friedman and Kruskal-Wallis tests. Spearman Correlation Coefficient was used for assessing the correlation between wear resistance and microhardness vaules for the restorative materials tested ($p<0.05$).

Results

According to the wear data evaluations (Table 2), the highest amount of wear was observed for the composite resin group (0,7538) which was followed by composite Cad/Cam block (0,4111), and hybrid Cad/Cam block (0,1484) groups, respectively. Among the composite blocks, Cerasmart showed the highest amount of wear (0,7518) and followed by Brilliant Crios (0,5499), HC block (0,3578), and Lava Ultimate (0,1974), respectively.

Table 2. Mean values of wear (µm) and microhardness(VHN) measurements.

		Wear Amount (µm)	Microhardness (VHN)
Composite Cad/Cam block	HC block	0,3578	76,2
	Cerasmart	0,7518	71,3
	Lava Ultimate	0,1974	97,9833
	Brilliant Crios	0,5499	77,5167
	Total	0,4111	77,5
Hybrid Cad/ Cam block	Vita Enamic	0,1484	185,1667
Composite Resin	IPS Empress Direct	0,7538	58,93

Regarding the microhardness measurements (Table 2), hybrid Cad/Cam block group showed significantly the highest VHN value (185,1667; p<0.001) and followed by composite Cad/Cam block (mean value of 77,5), and composite resin (58,93) groups, respectively. Among the composite Cad/Cam blocks, Lava Ultimate showed the highest VHN value (97,9833) and followed by Brilliant Crios (77,5167), HC block (76,2), and Cerasmart (71,3), respectively.

A significant correlation was determined between microhardness and wear resistance for the overall composite Cad/Cam blocks (p<0.001), whereas no correlation was observed for each composite block individually (p≥0.05). Also, no correlation was detected for hybrid Cad/Cam block and composite resin groups (p≥0.05). However, for the overall tested materials a significant correlation was found between microhardness and wear resistance (p<0.001) (Table 3, Figure 1).

Table 3. Corralation between wear and microhardness of the materials used in this study.

		Correlation Coefficient (r)	P (sig)
Composite Cad/ Cam block	HC block	-0.135	0.677
	Cerasmart	-0.18	0.956
	Lava Ultimate	-0.277	0.383
	Brilliant Crios	-0.277	0.383
	Total	-0.709	**0.001***
Hybrid Cad/Cam block	Vita Enamic	0.389	0.212
Composite Resin	IPS Empress Direct	0.203	0.526
	Total	– 0.83	**0.001***

* Positive correlation was found between microhardness and wear resistance (spearman correlation coefficient)

Discussion

This study investigated the correlation between surface hardness and wear resistance of six different restorative materials. As no correlation was observed for all the tested restorative materials individually, the null hypothesis was accepted.

The real performance of dental restorations should be assessed with long-term clinical observations, however this can be time consuming, expensive and arise ethical concerns (24). *In vitro* testing devices and methods were improved to overcome the mentioned problems. The chewing simulator, which was also used in the present study, was produced to mimic the oral environment *in vitro*. This simulator can evaluate both lateral and two body movement resistance of restorative materials (25). For the purpose to determine the proper parameters, researches were performed and different testing protocols were suggested by different authors (26,27). The testing parameters of the chewing simulator are loading forces, loading frequency, number of cycles, thermocycling and dry fatigue. The results of the studies were reported to be varying among these parameters (28). Teeth are exposed to substantial temperature changes during their intraoral functions. The temperature of dental enamel changes between 16°C to 48°C during these cycles (29). The alterations in temperature may lead to thermal stress and various modifications to dental hard tissues as well as the restorative materials as a result of different thermal expansion coefficients. Nelsen (22) reported that thermal characteristics of restorative materials could be examined by using thermocycling test method.

The Vickers microhardness test was used in this study to reflect mechanical properties of the materials and it was also suggested as an accurate and reliable method to measure surface hardness of materials, previously (28). Degree of polymerization, hydrolytic degradation, water absorption, inter-particle spacing and type, size, shape of inorganic fillers were determined as influencive factors affecting the wear resistance for restorative materials (30). Stawarczyk *et al*. reported that increase in filler loading and decrease in filler particle size improved the wear resistance (1). Klapdohr *et al.* reported a relation between inorganic filler content and hardness in resin-based composites (31). *Elzoheiry et al*. determined that, surface hardness was effected by filler particles and the link between polymer matrix and filler particles. The degree of polymerisation was also found to be related with the surface hardness (32).

The relation between the resistance to abrasion and microhardness was assessed for dental restorative materials in previous studies (1,22,28). CAD/CAM composite blocks were reported to have different mechanical properties due to their diversity in structural characteristics and filler percentages (33,34). Stawarcyzk *et al*. used composite blocks (Hc Block, Lava Ultimate, Cerasmart), a hybrid block (Enamic), and lithium disilicate glass ceramic and leucite (Ips Empress Cad) materials in their study, and aged in chewing simulator (5-55°C, 1.200.000 cycles, 50 N), in which human teeth were used as antagonist (26). According to their results, Enamic showed higher wear resistance compared to Lava Ultimate, Hc Block and Cerasmart block. Cao *et al* investigated two packable (Surefil, 3MP60, Dentsply, USA) two microhybrid (Clearfil AP-X, 3MZ250, Kuraray, Japan), and a nano-hybrid (Charisma Diamond, Kulzer, Germany) resin-based composites. A custom-made brushing machine was used to test the specimens (1Hz, 3N loads, 6×105 cycles). No significant difference was detected regarding interactions between wear and surface hardness for the materials (28). Dayan and Mumcu reported weak correlation between wear and microhardness of the restorative materials tested: Paradigm MZ100, 3M Espe, USA; Lava Ultimate, 3M Espe, USA; Vita Enamic, Vita Zahnfabric, Germany; Cerasmart, GC Corp., Japan (22). In the present study, supporting the results of Cao *et al.,* (28) *Dayan and Mumcu* (22), no correlation was observed for composite block, hybrid block and resin composite groups ($p \geq 0.05$). Also supporting the results of Stawarcyzk *et al*. (26), the hybrid ceramic Enamic group showed significantly the highest wear resistance among all groups ($p<0.001$). The filler volumes of tested materials were: Hc block: 61%, Cerasmart: 71%, Lava Ultimate: 80%, Brilliant Crios: 71%, Vita Enamic: 86%, IPS Empress Direct: 81,2%. The wear behavior of hybrid ceramic and composite materials might be associated with their different microstructures and filler contents. The ceramic or polymer content might have also affected the wear properites as well as mechanical and surface roughness, with regard to the fact that, decrease in surface hardness causes increase in the amount of wear (34).

Conclusion

Within the limitations of this study, the hybrid ceramic showed the highest wear resistance and microhardness, wheareas resin-based composite showed the lowest. According to our results, surface hardness may not be considered as a predictor for wear resistance of the restorative materials, however further studies should be undergone for more precise results.

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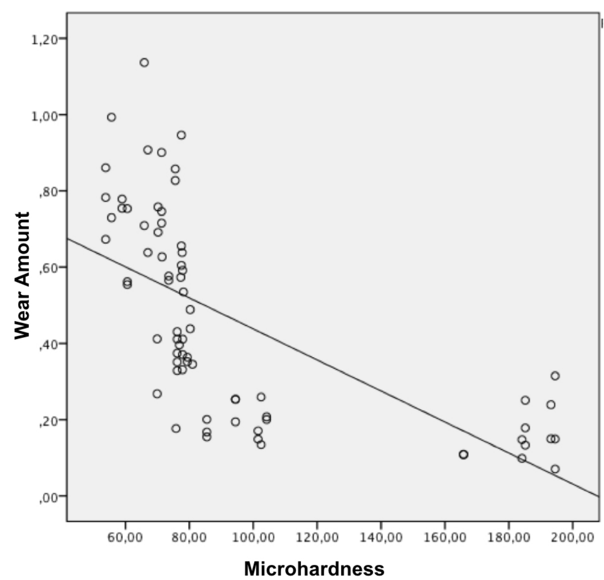

Figure 1. The relation between wear and microhardness

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