

## **In-vitro evaluation of wear characteristics, microhardness and color stability of dental restorative CAD/CAM materials**

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The aim of this study was to demonstrate the vertical and volumetric wear characteristics of CAD/CAM materials. The microhardness and color stability were evaluated. A polymer infiltrated ceramic network CAD/CAM block, resin nanoceramic CAD/CAM blocks, a resin composite, and enamel tissue were investigated. Samples were loaded in a chewing simulator. Other samples were aged and immersed in coffee. Color change was evaluated using the digital image analysis and a spectrophotometer. The data were analyzed using Kolmogorov-Smirnov, Kruskal Wallis, Mann Whitney *U*, Friedman, Spearman's rho tests ( $p < 0.05$ ). The lowest level of wear was obtained in enamel tissue group 0.20 (Q1:0.14; Q3:0.27 $\mu$ m). Resin composite group 2.48 (Q1:2.12; Q3:2.92) showed lower level of discoloration. No agreement was obtained between the digital image analysis and spectrophotometer data (Spearman's rho  $-0.314$ ,  $p = 0.014$ ). Similar wear rate to the natural enamel tissue was obtained for Vita Enamic. Digital image analysis was considered a promising technique for monitoring the color change.

**Keywords:** CAD/CAM, Discoloration, Microhardness, Composite, Wear

### INTRODUCTION

Restorative dentistry reinforced with a wide diversity of materials, systems and techniques for treatment of teeth in a minimally invasive way. Key factors in restoration success may be related to the proper application of the material and the selection of a precise technique<sup>1</sup>.

Resin composite materials can be applied with direct or indirect techniques. The choice of the technique is depending on numerous factors such as, the amount of remaining intact dental tissue following preparation, economic factors, time, and number of appointments. Direct technique allows restorations to be completed in a single visit. This technique has some disadvantages such as postoperative sensitivity, risk of secondary caries, and polymerization shrinkage, whereas the application of direct incremental technique may reduce the polymerization shrinkage. On the other hand, indirect technique requires more time and extra cost compared to the direct technique including various advantages of lower polymerization shrinkage, enhanced mechanical and physical properties of the restoration, better occlusal morphology, wear compatibility with natural dentition, and better proximal contacts<sup>2</sup>. The indirect technique can be carried out using either traditional materials or computer aided design and computer aided manufacturing (CAD/CAM) materials.

CAD/CAM systems have been improved since the Duret and Preston's research in 1970<sup>3</sup>. Three main components of the CAD/CAM systems are data acquisition, design, and manufacturing. Every step works independently from each other, and the proper application of each step is required to optimize the

results in production process. CAD/CAM systems can manufacture numerous materials including resin composites, metals, polymethyl methacrylate (PMMA), wax, resin nanoceramic (RNC), polymer infiltrated ceramic network (PICN), polycrystalline ceramics, and glass-ceramics.

The objective of using dental materials is to emulate enamel and dentin in terms of mechanical and biological structure however, it is quite difficult to re-create the complexity of the structure of natural dental tissues. Dental ceramics look similar to human enamel in terms of some physical properties, whereas composite resins simulate mainly the characteristics of dentin<sup>4</sup>. With regard to this information, PICN materials that combine ceramic and composite properties, were manufactured.

Ceramic or resin-based materials have been used in restorative dentistry, considering optical and mechanical properties, organic and inorganic content, and biocompatibility. Besides the clinical performance and capability of these materials are other determinants for the assessments to mimic dental tissues. Moreover, wear is another assessment criteria for clinical performance of dental materials<sup>5</sup>. Tooth wear has multifactorial etiology which is an irreversible and cumulative process. Physiological vertical loss of occlusal enamel wear was estimated to be 20–38  $\mu$ m, whereas pathological was 50  $\mu$ m annually<sup>6</sup>. Ideally, dental materials and enamel should have similar wear resistances. Accordingly, wear testing simulators are one of the most representative ways of clinical tooth wear with general agreement<sup>5</sup>. It is well known that microhardness of a material is correlated with resistance to intra-oral softening. Also, tea, coffee, acidic food, mouthwashes, and low pH drinks

may affect the surface hardness and color stability of resin-based materials. Color stability may limit the quality, longevity and aesthetic outlook of restorations<sup>7)</sup>. Discoloration of dental materials can be affected by staining drinks such as black tea, red wine, cola, and coffee which are common in daily diet. Coffee is one of the most effective beverages having high potential to stain<sup>8)</sup>. Composite resins and RNC CAD/CAM blocks may also be discolored by the staining solutions. Moreover, the color stability of composite resin materials can be influenced with the exposure time, composition of the restorative material, treatment of the restoration surface, and type of the staining solution<sup>9)</sup>.

The aim of this *in vitro* study was to evaluate the vertical/volumetric wear characteristics and the color stability of 4 RNC CAD/CAM blocks, a PICN CAD/CAM block, a conventional resin composite, and human enamel tissue, following one-year of thermocycled chewing simulation and coffee discoloration. Also, the correlation between the digital image analysis technique and a contact type clinical spectrophotometer device was assessed.

The ( $h_1$ ) hypotheses of the study were considered as follows: (1) wear rate of the tested restorative materials is different from enamel tissue, (2) color stability of the tested restorative materials and brands are different, (3) the Vickers microhardness (VHN) of the tested restorative materials are affected after coffee staining, (4) there is no correlation between the color measuring techniques.

## MATERIALS AND METHODS

Total of 6 different restorative materials including

a PICN CAD/CAM block; Vita Enamic (VITA, Bad Säckingen, Germany), 4 RNC CAD/CAM blocks; Hc block (Shofu, Kyoto, Japan), Cerasmart (GC, Tokyo, Japan), Brilliant Crios (Coltene Whaledent, Altstätten, Switzerland), Lava Ultimate (3M, St. Paul, MN, USA), a nanohybrid resin composite; IPS Empress Direct (Ivoclar Vivadent, Schaan, Liechtenstein), and natural human enamel tissue specimens (as control group) were investigated (Table 1).

### Specimen preparation

Total of 84 specimens were prepared for the wear test, containing 12 specimens per each group (a PICN CAD/CAM block group, 4 RNC CAD/CAM block groups, a resin composite group, and enamel tissue group). These specimens were embedded in acrylic resin (Imicryl, Konya, Turkey) and the top surfaces exposed for the wear test.

Enamel tissue group was included only for the wear test. Human maxillary central incisors were kept in % 0.1 of thymol solution for 6 months after the extraction till the study. The extracted teeth had no caries or restoration, and the reason for extraction was severe periodontal problems. Intact buccal surfaces of the maxillary central incisors were used for the enamel specimens. Then the enamel pieces were flattened by polishing with Sof-Lex discs (3M ESPE, Seefeld, Germany) in 4 different grains (55, 40, 24, and 8  $\mu$ m) respectively for 20 s at 20,000 rpm per each disc, under water cooling.

Another 120 specimens were prepared for the color analysis and microhardness tests, containing 20 specimens ( $n=10$  for coffee solution,  $n=10$  for control) per each group.

Table 1 The manufacturer and composition of the restorative materials

		Manufacturer	Monomer	Filler	
				Content	Weight%
Resin nanoceramic CAD/CAM block	Block Hc	Shofu, Kyoto, Japan	UDMA, TEGDMA	Silica-powder, zirconium silicate, micro fumed silica	61
	Cerasmart	GC, Tokyo, Japan	Bis-MEEP, UDMA, DMA	silica and barium glass nanoparticles	71
	Lava Ultimate	3M, St.Paul, MN,USA	Bis-GMA, UDMA, Bis-EMA, TEGDMA	Silica and zirconia nanoparticles and zirconia/silica nanoclusters	80
	Brilliant Crios	Coltene Whaledent, Altstätten, Switzerland	Bis-GMA, Bis-EMA, TEGDMA	Barium glass and silica particles	71
Polymer infiltrated ceramic network CAD/CAM block	Vita Enamic	Vita Zahnfabrik, Bad Säckingen, Germany	UDMA, TEGDMA	Polymer-infiltrated-feldspatic ceramic-network material	86
Resin composite	IPS Empress Direct	Ivoclar Vivadent, Schaan, Liechtenstein	Dimethacrylate	Barium alumina fluorosilicate glass, barium glass filler, mixed oxide, ytterbium trifluoride	81.2

The specimens of CAD/CAM blocks were prepared using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) in 3 mm of thickness under continuous water-cooling. The surfaces of the specimens were polished with P 400, P 600, P 800, P 1000 silicon carbide abrasive papers, respectively, under running water (170 rev/m for 15 s; Minitech 233, Presi, Grenoble, France). The abrasive papers were renewed for every 10 samples. The polishing procedure was performed by one operator.

Composite resins were placed incrementally (2+1 mm) in 3 mm thickness silicone molds under finger pressure using mylar strips and light-cured for 20 s with a LED curing unit (Kerr Demi Ultra, Kerr, Orang, CA, USA). Sof-Lex Discs in 4 different grains (coarse, 100 µm; medium, 40 µm; fine, 24 µm; and super-fine, 8 µm Al<sub>2</sub>O<sub>3</sub> particles) were used sequentially for both surfaces under running water for surface polishing (20 s application at 20,000 rpm, per disc).

#### Mechanical tests

Eighty-four wear test specimens were loaded in a chewing simulator (Dent Ar-Ge, Analitik Medical, Turkey), and undergone for 240,000 chewing cycles against a stainless steel ball with a diameter of 3 mm. Parameters of chewing simulator were set at, 49 NF, 1.7 Hz frequency, 1.5 mm lateral movement with termal cycling, 5°C–55°C cold and hot bath temperature, 60 s waiting time, and approximately 1,800 thermocycles were loaded. Three dimensional morphologies of the worn surfaces were scanned with a Laser Scanner (Las-20, SD Mechatronic, Munich, Germany) and the collected data were recorded. Vertical and volumetric wear amounts were calculated using a specific software programme (Geomagic Control, 3D Systems, Rock Hill, SC, USA) using the three-point alignment method. Three measurements were done for each sample and the avarage value was taken as the reference.

The other 120 specimens for color analysis and microhardness tests were aged using 1,500 thermocycles (THE-1100, SD Mechatronic), between 5°C–55°C and dwell time of 15 s. Ten specimens from each group were immersed in coffee solution and kept at 25°C room temperature for 7 days. The solution was generated by adding 30 gr of coffee (Nescafe Gold, Nestle, Vevey, Switzerland) into 100 mL of distilled water (pH of coffee is 5.00)<sup>10</sup>. Ten specimens from each group were assigned as the control group, which were also kept in distilled water for 7 days at 25°C room temperature. The coffee solution was renewed daily<sup>9</sup>. VHN was assessed for the baseline and after the aging and discoloration procedures (under 200 gr force for 10 s).

#### Optical test

Initial shade determination was performed using a clinical contact-type spectrophotometer (VITA Easy Shade, VITA Zahnfabrik) and the digital image analysis method (Photoshop CC, Adobe Systems, San Jose, CA, USA). The shade determination was repeated following the discoloration procedures using both techniques.

#### Assessment of the color

Regarding the spectrophotometer measurements, *L\**(Lightness), *C\**(Chroma) and *H\**(Hue) parameters were recorded for each specimen. The color changes ( $\Delta E_{00}^*$ ) were calculated using a specific formula based-on CIEDE 2000 color space<sup>11</sup>:

$$\Delta E_{00}^* = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C}\right) \left(\frac{\Delta H'}{K_H S_H}\right)}$$

Regarding the digital image analysis measurements, Mobile Dental Photography device (Smile Lite MDP, Smile Line, St-Imier, Switzerland) was used to obtain standardized dental photographs in terms of light and distance. *L\**(Lightness), *A\**(red-green hue), *B\**(yellow-blue hue) parameters were collected using Photoshop CC software for each photograph. The color changes ( $\Delta E_{00}^*$ ) were calculated using another specific formula based-on CIEDE 2000 color space.:

Following the discoloration procedures, the threshold  $\Delta E^*$  value for clinical perceptibility was considered as  $\geq 0.8$  and acceptability was considered as  $1.8^{12}$ .

#### Statistical analysis

Statistical analysis was performed using a statistical software (SPSS Version 23, SPSS, Chicago, IL, USA). Data conformity was evaluated with Kolmogorov-Smirnov test and pairwise groups were tested using Mann Whitney *U* test. In order to compare more than two independent groups, Kruskal Wallis test was used. Multiple comparisons of the groups were performed with Friedman test. Spearman's rho was used for the qualified reliability of the analysis for spectrophotometer and digital image analysis techniques. The significance level was set at  $p < 0.05$ .

## RESULTS

In terms of the assesments of wear resistance between the groups, following the 240,000 chewing cycles, the enamel tissue group showed the greatest wear resistance ( $p < 0.001$ ) and followed by PICN CAD/CAM block, RNC CAD/CAM block and resin composite groups, respectively. The highest vertical peak was observed for the IPS Empress Direct (0.97 µm) composite, whereas the lowest was for the enamel tissue (0.165 µm; Table 2)

Both the brands ( $p < 0.001$ ) and the restorative material ( $p < 0.001$ ) were considered effective factors regarding the level of volumetric wear (Table 3). IPS Empress Direct showed the greatest level of volumetric wear median 0.75 (Q1:0.70; Q3:0.82) and followed by Cerasmart, Hc Block, Brilliant Crios, Lava Ultimate, Vita Enamic materials, and enamel tissue, respectively (Table 3). There was no significant difference for the volumetric wear measurements between Cerasmart and IPS Empress Direct ( $p = 1.000$ ), between HC block and Brilliant Crios ( $p = 0.073$ ), between Lava Ultimate and Vita Enamic ( $p = 0.987$ ) between Lava Ultimate and Enamel tissue ( $p = 1.000$ ; Table 4). PICN ceramic CAD/CAM block group (Vita Enamic) showed the closest

Table 2 The maximum amount of vertical loss regarding the restorative materials and the brands

Restorative material	Brand	Vertical peak ( $\mu\text{m}$ )
Resin nanoceramic CAD/CAM Block	HC Block	0.564
	Cerasmart	0.368
	Lava Ultimate	0.203
	Brilliant Crios	0.280
		0.564
Polymer infiltrated ceramic network CAD/CAM Block	Vita Enamic	0.203
Resin Composite	Ips Empress Direct	0.970
Enamel Tissue	—	0.165
$p<0.001$	$p<0.001$	0.970

Table 3 Volumetric wear amounts of the restorative materials and the brands

Restorative material	Brand	Wear amount (volume) ( $\mu\text{m}$ )		
		Q1	Median	Q3
Resin nanoceramic CAD/CAM Block	Hc Block	0.33	0.36	0.40
	Cerasmart	0.67	0.76	0.89
	Lava Ultimate	0.16	0.19	0.22
	Brilliant Crios	0.43	0.54	0.59
		0.26	0.41	0.64
Polymer infiltrated ceramic network CAD/CAM Block	Vita Enamic	0.11	0.15	0.18
Resin composite	Ips Empress Direct	0.70	0.75	0.82
Enamel Tissue	—	0.19	0.37	0.65
$p<0.001$	$p<0.001$			

Table 4 Multiple comparisons of volumetric wear amounts regarding the brands

Brand	Brand	Mean difference ( $\mu\text{m}$ )	$p$
Cerasmart	HC Block	0.409	<0.001
	Lava Ultimate	0.575	<0.001
	Brilliant Crios	0.269	<0.001
	Ips Empress Direct	-0.003	1.000
	Vita Enamic	0.608	<0.001
	Enamel Tissue	0.550	<0.001
HC Block	Lava Ultimate	0.166	<0.001
	Brilliant Crios	-0.139	0.073
	Ips Empress Direct	-0.412	<0.001
	Vita Enamic	0.199	<0.001
	Enamel Tissue	0.141	0.036
Lava Ultimate	Brilliant Crios	-0.306	<0.001
	Ips Empress Direct	-0.578	<0.001
	Vita Enamic	0.032	0.987
	Enamel Tissue	0.024	1.000
Brilliant Crios	Ips Empress Direct	-0.272	0.004
	Vita Enamic	0.339	<0.001
	Enamel Tissue	0.281	<0.001
Ips Empress Direct	Vita Enamic	0.611	<0.001
	Enamel Tissue	0.553	<0.001
Vita Enamic	Enamel Tissue	0.057	0.979

volumetric wear value 0.15 (Q1:0.11; Q3:0.18) to natural enamel tissue 0.20 (Q1:0.14; Q3:0.27), which two values were also statistically similar ( $p=0.670$ ; Table 5).

In terms of the VHN values, regarding the baseline measurements, the highest value was obtained for Vita Enamic control 259 (Q1:253.9; Q3:286.3) and the lowest for IPS Empress Direct control 60 (Q1:57.1; Q3:66.9) groups (Table 6). All groups showed significant differences for the Vickers hardness values as well

as microhardness values regarding the comparisons between baseline values and the values after coffee discoloration ( $p^1$  refers to the relation between baseline coffee solution and coffee solution groups;  $p^2$  refers to the relation between baseline control and control groups; Table 6). Regarding  $p^1$  and  $p^2$ , the greatest level of decrease for microhardness was obtained for Vita Enamic and the lowest was for Ips Empress Direct. Regarding the correlation between coffee solution and

Table 5 Multiple comparisons of volumetric wear amounts regarding the restorative materials

Restorative material	Restorative material	Mean difference ( $\mu\text{m}$ )	$p$
Resin nanoceramic CAD/CAM Block	PICN CAD/CAM Block	0.294	<0.001
	Resin composite	-0.316	<0.001
	Enamel Tissue	0.237	<0.001
Polymer infiltrated ceramic network CAD/CAM Block	Resin composite	-0.611	<0.001
	Enamel Tissue	0.057	0.670
Resin composite	Enamel Tissue	0.553	<0.001

Table 6 Vickers microhardness measurements of the samples

Restorative material	Brands	Baseline coffee solution group			Coffee solution group			Baseline control group			Control group			$p^1$	$p^2$	$p^3$
		Q1	Median	Q3	Q1	Median	Q3	Q1	Median	Q3	Q1	Median	Q3			
Resin nanoceramic CAD/CAM block	HC Block	112.7	117.8	128.1	73.10	77.3	85.60	91.9	95.9	99.4	80.1	85	91.50	<0.001	0.009	0.116
	Cerasmart	82.9	85.9	87.7	69.3	72.6	76	92.5	97.9	111.7121	75.9	81.80	85.30	0.004	0.039	0.296
	Lava Ultimate	115.9	120.3	128.4	90.5	98.10	102	109	113	89.5	98.8	103	109.7	0.001	0.002	0.021
	Brilliant Crios	85.9	90.2	94.2	69.20	74.10	78.70	83.5	86.8	89.5	77.6	79.50	83.1	0.004	0.010	0.160
			86.9	98.8	120.2	71.70	76.90	86.15	87.6	98.5	121.1	79.43	84.85	98	<0.001	<0.001
Polymer infiltrated ceramic network CAD/CAM block	Vita Enamic	222.5	237.9	272	171	189.10	195	253.9	259	286.3	189.1	205.6	217.6	0.006	0.007	0.386
Resin composite	Ips Empress Direct	59.2	63.4	69.7	54	58.10	62.8	57.1	60	66.9	52.1	56.6	60.9	0.007	0.012	0.747

$p^1$ : Statistical relation between, baseline coffee solution group and coffee solution group

$p^2$ : Statistical relation between, baseline control group and control group

$p^3$ : Correlation between, coffee solution group and control group

Table 7 The assessments of the spectrophotometer measurements regarding  $\Delta E^*$  values

Restorative material	Brand	$\Delta E^*$ Spectrophotometer			$\Delta E^*$ Digital image analysis		
		Q1	Median	Q3	Q1	Median	Q3
Resin nanoceramic CAD/CAM Block	HC Block	1.35	1.74	2.64	3.19	4.30	5.28
	Cerasmart	2.37	2.91	3.86	1.90	3.60	5.88
	Lava Ultimate	2.17	2.44	2.70	4.51	5.34	7.06
	Brilliant Crios	2.83	3.76	4.60	2.53	3.57	4.03
		2.16	2.65	3.04	3.13	4.08	5.53
Polymer infiltrated ceramic network CAD/CAM Block	Vita Enamic	2.47	3.74	4.26	1.87	3.24	4.48
Resin Composite	Ips Empress Direct	2.12	2.48	2.92	3.19	3.50	4.58
				$p=0.004$			$p=0.011$

control groups ( $p^3$ ), only Lava Ultimate values were statistically significant ( $p=0.021$ ; Table 6).

Regarding the spectrophotometric analyses, resin composite group showed perceptible and the highest ( $4.2\pm 1.8$ )  $\Delta E^*$  value and followed by (RNC) CAD/CAM block group ( $3.5\pm 1.4$ ), which was also perceptible, and PICN group ( $3.2\pm 0.9$ ), which was imperceptible, respectively (Table 7). However, brand ( $p=0.390$ ) and restorative material ( $p=0.618$ ) were not considered to be effective factors for the amount of color change after discoloration (Table 7).

Regarding the spectrophotometric analysis, resin composite group showed the lowest 2.48 (Q1:2.12; Q3:2.92)  $\Delta E$  value followed by (RNC) CAD/CAM block group 2.65 (Q1:2.16; Q3:3.04) and PICN block group 3.74 (Q1:2.47; Q3: 4.26) (Table 7). Restorative material ( $p=0.160$ ) was not considered to be as an effective factor for the amount of color change after discoloration, however the brand was effective ( $p=0.004$ ) (Table 7). A negative correlation (Spearman's rho  $-0.314$ ) was found between the spectrophotometer and Photoshop measurements and the relationship was statistically significant ( $p=0.014$ ).

## DISCUSSION

According to the results of the current study, the level of wear for Vita Enamic and Lava Ultimate materials were statistically similar compared to the enamel tissue ( $p=0.979$  and  $p=1.000$ , respectively; Table 4). The amount of color changes for the tested restorative materials was not statistically significant ( $p=0.160$ ). The Vickers hardness values concerning the comparisons between the baseline values and the values after coffee staining, were statistically significant among all groups (Table 6). Also, the two-color measurement techniques used in this study were correlated negatively (Spearman's rho  $-31.4\%$ ).

The most reliable way to evaluate dental restorative materials is the long-term clinical trials, however, during the pandemic of COVID-19, became difficult to management of clinical studies. On the other hand, chewing simulators are one of the *in vitro* simulation devices used for the wear testing. Numerous researchers have studied on multiple parameters, in order to simulate the clinical situations. The magnitude of force, number of cycles, loading frequency, vertical and lateral movements are assigned as the loading parameters. Loading force was considered as a significant parameter, effecting the loaded object for fatigue<sup>13</sup>. The number of cycles range from 10,000 to 3.6 million cycles (0.04–14.4 clinical years), and 240,000 stress cycles were determined to simulate one year period, clinically<sup>14</sup>. In this study, the specimens were aged with 240,000 chewing cycles to mimic one-year of clinical aging. In the present study, 1.7 Hz frequency was considered for the masticatory frequency of the chewing simulator device, regarding the previously reported physiologic masticatory frequency for humans as 1 to 2 Hz<sup>15</sup>.

One of the best methods for measuring wear was

considered to be the three-dimensional (3D) images of materials<sup>16</sup>. 3D images are scanned using numerous methods such as non-contact laser scanners, micro- or cone- CT scanners, and contact profilers. 3D scanning is feasible to both laboratory and clinic, accurate and quantitative method, and it also provides storable database, however, it needs software and hardware costs<sup>17</sup>. LAS 20-laser scanner is one of the scanning devices providing 3D image processing. Almost all materials can be analyzed using LAS 20 laser scanner and many sensor parameters can be specialized by users. In this study, Las 20 laser scanner device was used for capturing the worn surfaces. Following that, vertical and volumetric wear amounts were calculated using a specific software programme (Geomagic Control, 3D Systems).

Long term water storage or thermocycling are the most common artificial aging techniques reported in literature<sup>17</sup>. The International Organization for Standardization (ISO) declares that the appropriate water degrees should be between 5°C and 55°C for the aging tests, according to TR 11450 standards<sup>17</sup>. Accordingly, in our study the thermocycling degrees were set at 5°C and 55°C. Also, the specimens were immersed in coffee solution for 1 week referring to the literature of that, 1 week of coffee immersion is equivalent to 7 months of clinical usage<sup>18</sup>.

Discoloration of composite materials can be measured by instrumental or visual techniques<sup>7</sup>. Visual technique was not considered a precise method because of the distinction in color perception<sup>19</sup>. Whereas instrumental technique is a more reliable method for the color assessment<sup>20</sup>, which can be performed using image analysis with dental photography or digital shade taking devices such as colorimeter and spectrophotometry. Image analysis<sup>21-23</sup> and spectrophotometric analysis<sup>20-24</sup> techniques have been used in several studies previously. In the present study, clinical contact-type spectrophotometer device and image analysis techniques were the two methods used for evaluating the shade selection, quantitatively.

### Assessment of the wear

The range of wear amounts of the materials used in this study were considered between 0.20 (Q1:0.14; Q3:0.27  $\mu\text{m}$ ) and 0.75 (Q1:0.70; Q3:0.82) (Table 3). Vita Enamic was reported to have a higher level of microhardness and elastic modulus than the resin-based materials and also presented the closest surface hardness values to dental enamel tissue, previously<sup>25,26</sup>. Supporting these findings, according to our results, Vita Enamic and Lava Ultimate materials presented similar microhardness values to the dental enamel. Therefore, the 1st hypothesis was rejected.

The wear of a dental material is related to the composition and characteristics of the material, and roughness properties of polymer or ceramic-based materials included. Accordingly, Vita Enamic exhibited comparable wear resistance to the enamel tissue group, whereas (RNC) CAD/CAM blocks showed significantly

lower wear resistance among all. This might be as a result of addition of ceramic particles into the polymer matrix (PICN, RNC)<sup>5)</sup>. Vita Enamic is a PICs and Lava Ultimate, Brilliant Crios, Hc Block and Cerasmart are RNCs<sup>27)</sup>. Schlenz *et al.*<sup>28)</sup> have studied 3 (RNC) CAD/CAM blocks (Lava Ultimate; Brilliant Crios; Cerasmart), a PICN block (Vita Enamic), and a lithium disilicate ceramic block (IPS E.max CAD) *in vitro*, previously. The materials were aged in a chewing simulator and the wear amounts were measured using a digital microscope. Vita Enamic showed higher wear resistance than Brilliant Crios and Cerasmart materials, which was incompatible with our results<sup>28)</sup>.

In the present study, Lava Ultimate showed similar amount of volumetric wear with Vita Enamic with no significant difference ( $p=0.987$ ). There are some studies in the literature supporting and opposing to our results. The results of Matzinger *et al.*<sup>29)</sup> and Tsujimoto *et al.*<sup>30)</sup> support our results, whereas the results of Lawson *et al.*<sup>31)</sup> and Lauvahutanon *et al.*<sup>32)</sup> oppose to. According to Lawson *et al.*<sup>31)</sup> and Lauvahutanon *et al.*<sup>32)</sup>, Vita Enamic have a higher level of volumetric wear compared to Lava Ultimate.

According to literature, the (RNC) materials containing larger filler particles (*i.e.* Lava Ultimate) present higher volumetric wear amounts. On the contrary the materials containing smaller filler particles (*i.e.* Brilliant Crios) present condensable characteristics and lower volumetric wear amounts<sup>29)</sup>. Opposing to these findings, in the present study, the Brilliant Crios presented higher volumetric wear than the Vita Enamic. The filler content of these materials might be the explanation of this result (Vita Enamic, wt: %86; Brilliant Crios, wt: %71)<sup>5)</sup>. Accordingly, there are several results supporting that wear is a complicated element with various parameters, directly related to the design of the study.

Nguyen *et al.* reported that high-pressure and high-temperature (HP/HT) polymerization (300 MPa, 180°C) of CAD/CAM blocks may provide higher physical and mechanical properties than resin composites<sup>33)</sup>. Supporting Nguyen *et al.* the resin composite group showed significantly the highest wear among all groups in our study, which might be associated with the do not performing HP/HT polymerization. In another study, Gwon *et al.*<sup>34)</sup> have evaluated the wear properties of opposing CAD/CAM materials and posterior resin-based composite materials, using a chewing simulator. Resin-based composites (MI Gracefil, GC; Gradia Direct P, GC; Estelite  $\Sigma$  Quick, Tokuyama Dental, Tokyo, Japan; Filtek Supreme Ultra, 3M ESPE) presented considerable weight loss compared to the CAD/CAM blocks (Zirkonzahn Prettau, Zirkonzahn, Bruneck, Italy; Rosetta SM, HASS, Kangneng, Korea; Rosetta SM, HASS). This result is also compatible with our results, as the resin composite group have showed the highest wear amount among all materials.

#### Assessment of the color change

In the present study, the  $\Delta E$  values were not

statistically significant among the restorative materials but they were significant for the brands. Therefore the 2nd hypothesis was partially rejected.

Although Acar *et al.*<sup>7)</sup> reported that the color change levels were different for the tested CAD/CAM and nanocomposite resin materials, there are also studies with opposite results. Alharbi *et al.*<sup>35)</sup> evaluated the optical properties of CAD/CAM blocks and resin composite materials, and no color change was reported for the tested materials.

Thereby, optical properties of the materials might be affected by mechanical properties of the materials, which is mainly associated to the filler content. Higher hardness values were detected in specimens with higher filler contents, and they reported that there is a correlation between microhardness and the color stability<sup>36)</sup>.

Regarding the perceptibility of the  $\Delta E$  values, all of the groups showed perceptible ( $\geq 0.8$ ) and not acceptable ( $\geq 1.8$ ) level of color changes. IPS Empress Direct group showed higher color change than Lava Ultimate and HC block (Table 7). The discoloration sensitivity of the resin composites may depend on the hydrophilicity of organic matrix and the level of water absorption. The level of water intake by the resin matrix, is attributed to the bond between the filler and the resin content of the material<sup>37)</sup>. If the material is able to absorb water, it can also absorb other colored liquids, which may lead to discoloration. As the hydrophilicity of the resin composites are higher than the ceramics<sup>9,35)</sup>, they have higher affinity to the staining solutions<sup>38)</sup>.

#### Assessment of microhardness

The measurements were statistically different between the baseline values and the values after coffee discoloration (Table 6). Microhardness values gradually decreased between the baseline values and the values after coffee discoloration for all groups. Therefore, the 3rd hypothesis was accepted.

Although the pH of coffee is 5.00<sup>10)</sup>, as it dissolves in water, the impact of this water intake may damage the polymer matrix. Following the absorption of water from polymer ingredients, the coupling agents may hydrolyze and adversely affect the chemical bond between resin matrix and the filler particles. Finally, the filler particles may remove from the material surface and thereby surface hardness may decrease. As a result, the immersion in coffee solution might have an adverse effect on the surface microhardness in the present study. In addition, the effect on inorganic fillers and resin-filler interface might have influenced the decrease in surface hardness, through expansion of the network and decrease in frictional forces among polymer chains<sup>39)</sup>. It may explain why resin-based materials show lower hardness values after thermocycling (Table 6). This might also be the explanation of lower surface hardness of resin-based materials after exposure to beverages.

In the present study, Vita Enamic showed the highest baseline Vickers Hardness values, which can be related to the ceramic network structure of this material<sup>40)</sup>. Vita

Enamic contains a polymer-infiltrated feldspar ceramic network, reinforced with aluminum oxide, and in this way it may assemble the useful features of both ceramic and composites<sup>41</sup>. Dayan and Mumcu<sup>40</sup>, Lawson *et al.*<sup>31</sup>, and Koizumi *et al.*<sup>41</sup>, reported that Vita Enamic have higher surface microhardness values than the Cerasmart, and this was explained by the higher resin content of the Cerasmart material (29%). According to our results, supporting these previous studies, Vita Enamic showed higher microhardness values compared to the Cerasmart (Table 6). Lava Ultimate showed the higher baseline Vickers Hardness values than the Hc Block. It was previously reported that, the higher filler content, the higher surface hardness value<sup>42</sup>. Thus, our result might be as a result of higher inorganic filler content of Lava Ultimate (80 wt%), compared to the Hc Block (61 wt%). Additionally, Brilliant Crios showed lower baseline Vickers Hardness values than Lava Ultimate. The filler size has also an effect on mechanical properties of (RNC) materials, a greater filler size provides greater hardness, flexural modulus, and flexural strength, whereas smaller filler particle size may induce a smooth surface<sup>43</sup>. The greater size of the fillers in Lava Ultimate (20 nm) than the Brilliant Crios (<20 nm) might be the reason of higher microhardness values for Lava Ultimate. The Lava Ultimate blocks comprise silica (20 nm) and zirconia (4–11 nm) nano-particles, which together form nano-clusters in 0.6–10  $\mu\text{m}$ . This combination also provides a structural integrity and allows a greater amount of ceramic fillers incorporated<sup>44</sup> besides, interstitial gaps between the particles are filled with nanomers and this construction provide higher ceramic content.

Cerasmart showed lower baseline Vickers Hardness values than Hc block, in spite of a higher filler content (Cerasmart, wt: 71%; Hc Block, wt: 61%). TEGDMA molecule size is lower than Bis-GMA and UDMA molecules. Thanks to the molecule size of TEGDMA, it includes higher double bonds concentration and shows the higher crosslink density, provide form the tighter networks. Therefore, TEGDMA contribute to enhance (RNC) materials surface hardness. The TEGDMA content in Hc Block might be the explanation of this result<sup>45</sup>.

#### *Correlation between the color measuring techniques*

Spectrophotometric and digital image processing measurements were not correlated (Spearman's rho  $-0.314$ ,  $p=0.014$ ; Table 7). Thus, 4th hypothesis was accepted. The digital image analysis method might be considered as a reliable method for the shade selection of dental materials. However, the *in vitro* studies regarding this topic in the literature are controversial<sup>20-23</sup>. Anand *et al.*<sup>21</sup>, McLaren *et al.*<sup>22</sup>, Zhang and Yu<sup>23</sup> have considered digital image analysis as a reliable method, whereas Farah<sup>24</sup> also found a good agreement on  $\Delta E^*$  measurements between spectrophotometer and digital image analysis methods, nevertheless spectrophotometer measurements were considered more reliable.

Digital image analysis method can be affected by

camera and objective type, shooting parameters, flash light, cross polarization (CP) filter, and the selected computer software. Thus, in order to obtain appropriate digital images, standardization should be provided<sup>22</sup>. Shooting distance according to the selected camera and objective, shutter speed, aperture, white balance, and ISO should be determined in appropriate conditions especially for shade analysis<sup>46</sup>. Shade selection by using spectrophotometer is considered as a gold standard on the other hand the digital image analysis is the novel method for the shade analysis. According to the results of the current study, digital image analysis technique does not appear to be convenient for shade selection, but further clinical evaluations are needed.

The use of only a single material in the PICN CAD/CAM block group can be considered is a limitation of this *in vitro* study. Clinical evaluations with expanded materials should be designed for further studies.

## CONCLUSION

The highest wear rates were obtained for resin composite materials and the lowest for enamel tissue. Vita Enamic and Lava Ultimate were found to have the closest wear value to the enamel tissue. The lowest level of discoloration was obtained for resin composite materials. Vita Enamic presented the highest Vickers Hardness values. Digital image analysis may not be considered as a reliable method for shade selection of dental materials.

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