

The Effect of Heating on Surface Microhardness of Resin-based Materials for Direct Restoration

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The aim of this study was to evaluate and to compare the surface hardness of three types of resin-based materials used for direct restoration after heating at different temperatures. A giomer (Beautiful II, Shofu Dental), a compomer (Dyract eXtra, Dentsply Sirona) and a hybrid composite resin (Gaenial Posterior, GC Corporation) were selected for this study. Twenty disk-shaped specimens of each material were heated at room temperature (21°C), at 37°C, at 50°C and at 60°C. Vickers microhardness test was performed on top and bottom surfaces using digital microhardness tester (Micro-Vickers Hardness System CV- 400DMTM, CV Instruments Namicon). The top and bottom surfaces VHN was calculated as a mean value of five determinations. Also, the microhardness ratio was calculated by dividing the top mean VHN value by bottom mean VHN value. Increased mean hardness values were recorded after heating, irrespective of resin-based tested materials. The highest hardness values were recorded after heating all three materials at 60°C, followed by the hardness recorded at 50°C, 37°C and 21°C. For top surfaces, the lowest hardness value was recorded in Dyract eXtra group when samples were warmed at room temperature and the highest hardness value was obtained in Beautiful II group when samples were heated at 60°C. For the bottom surfaces, Dyract eXtra specimens heated at 21°C presented the lowest hardness values and Beautiful II samples heated at 60°C presented the highest hardness values. On top and on bottom surfaces Dyract eXtra showed the lowest hardness values, followed by G-aenial Posterior and Beautiful II, irrespective the heating temperature.

Keywords: Vickers hardness, compomer, giomer, composite resin

Composite resins have gain popularity during the time and now are the most used materials for direct restoration. Unfortunately, in oral environment, these materials are exposed to different chemical and physical aggressions [1, 2] and are prone to degradation [3]. Dental practitioners have claimed some disadvantages of these materials like polymerization shrinkage, microleakage, postoperative sensitivity, poor marginal adaptation, sensitive technique for placement, difficult handling. The producers made serious effort to improve also mechanical properties of composite resins. It was demonstrated in previous studies that pre-heating before photoactivation decreases material viscosity, which might lead to lower microleakage [4] and better marginal adaptation [5]. Increased surface hardness and enhanced flexural and diametral tensile strength were also reported as a result of overall conversion [6, 7]. Due to increased temperature by pre-heating the monomer and radical mobility will be enhanced and the degree of conversion will be higher [8-11]. As a result of higher double bond conversion, increased volumetric shrinkage [12, 13] and greater shrinkage stress will result during polymerization.

New alternative hybrid direct restorative materials were introduced on the market in the last decades. Polyacid modified composites or compomers have dimethacrylate monomer having two carboxylic groups present in their structure and fillers that are similar to the ion-leachable glass present in glassionomer cements. They have some advantages like dentin and enamel adhesion, stable matrix structure, fluoride releasing and reduced microleakage [14-17]. Giomers are resin composite with innovative, pre-

reacted glassionomer fillers [18]. The inorganic fillers derive from complete or partial reaction of ion-leachable fluoroaluminosilicate with polyalkenoic acids [19]. Some advantages were mentioned for this material: fluoride release and recharge, enhanced mechanical, esthetical and handling properties [20, 21]. Lack of knowledge is in the literature regarding the mechanical properties of these hybrid materials when they are preheated. The evaluation of warmed restorative materials mechanical properties is essential to determine the effect of heat on material capability to resist to occlusal forces, fracture and wear. Surface hardness, compressive strength [22], and diametral tensile strength are common investigations to establish the mechanical behavior of restorative materials in oral cavity [23-25].

The aim of this study was to evaluate and to compare the surface hardness of three types of resin-based materials used for direct restoration after heating at different temperatures.

Experimental part

Specimen's preparation

Three resin-based materials: a giomer (Beautiful II, Shofu Dental) (group 1), a compomer (Dyract eXtra, Dentsply Sirona) (group 2) and a hybrid composite resin (Gaenial Posterior, GC Corporation) (group 3) were selected for this study. Details regarding their composition are presented in table 1.

Twenty disk-shaped specimens of each material were obtained using metal molds having 10 mm in diameter and 2 mm in thickness. The specimens were randomly

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Table 1
CHEMICAL COMPOSITION OF RESIN-BASED MATERIALS INCLUDED IN THE STUDY

Material for restoration	Filler	Particle size range (µm)	Monomer	Manufacturer
Beautiful II	Multi-functional glass filler S-PRG filler (fluoroboroaluminosilicate glass)	0.01-4.00	Bis-GMA, TEGDMA	Shofu Dental
Dyract eXtra	Strontium fluoride glass	0.10-1.40	Bis-GMA, TEGDMA TMPTMA	Dentsply Sirona
Gaenial Posterior	Pre-polymerized fillers: strontium, strontium and lanthanoid fluoride, fluoroaluminosilicate	16.0-17.0	UDMA, dimethacrylate co-monomers	GC Corporation

and equally divided in three subgroups. In subgroup 1 the specimens were preheated at room temperature (21°C), in subgroup 2 the specimens were preheated at 37°C, in group 3 the specimens were preheated at 50°C and in group 4 the specimens were preheated at 60°C. In order to obtain a homogeneous warming process and to avoid multiple heating, unidose capsules instead of syringe were used. The capsules of the each material were heated at the specified temperatures for 30 min in a dry-bath incubator (Labline Equipment PVD LTD). A standard time of 30 s was established to transport the capsules from the incubator and to start specimen preparation. In order to obtain the specimens, the mold was placed on a glass slab, in direct contact to a mylar strip. One layer of each material was condensed into the mold, than the mold was covered with a mylar strip and pressed with a glass slide to remove the excess of the material. The layer of resin-based materials has been polymerized for 40 seconds using a LED light unit (LED B, Guilin Woodpecker Medical Instrument Co., Ltd, China) having the light intensity of 850-1000mW/cm² and the wavelength of 420-480 nm. The specimens were polished with 600, 800 and 1200 grit silicon carbide paper under water cooling and than stored in distilled water [19] in a dark recipient at 37°C for 24 h.

Microhardness determination

Vickers microhardness test was performed on top and bottom surfaces using digital microhardness tester (Micro-

Vickers Hardness System CV- 400DMTM, CV Instruments Namicon). A 50 g load was applied through a Vickers indenter. Five indentations were made for each specimen. A standardized distance of 1 mm was maintained between two successive indentations. The microhardness was determined by measuring the lengths of the diagonals of the indentation marks and expressed as a Vickers Hardness Number (VHN). The top and bottom surfaces VHN was calculated as a mean value of five determinations. Also, the microhardness ratio was calculated by dividing the bottom VHN value by top VHN value.

Statistical analysis

Wilcoxon statistical test was used to compare the top and bottom hardness of each material after heating at different temperatures, the hardness values of different materials heated at the same temperature and the hardness ratio of the materials at different temperatures.

Results and discussions

The mean Vickers microhardness values (VHN) on the top and on the bottom surfaces of three commercial resin-based materials pre-heated at different temperatures are presented in table 2.

Increased mean hardness values were recorded after heating, irrespective of resin-based tested materials. The highest hardness values were recorded after heating all

Table 2
MEAN VICKERS MICROHARDNESS VALUES (VHN)

Material	21°C		37°C		50°C		60°C	
	Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom
Dyract eXtra	54.24 ± 1.34	44.93 ± 0.99	56.05 ± 0.48	46.56 ± 0.60	63.58 ± 0.77	48.23 ± 0.70	68.83 ± 0.45	50.37 ± 0.67
Beautiful II	62.74 ± 1.11	52.42 ± 0.94	65.83 ± 0.47	54.65 ± 0.44	69.42 ± 0.80	58.63 ± 0.40	74.68 ± 0.44	61.34 ± 0.31
G-aenial Posterior	57.81 ± 0.42	49.54 ± 0.34	60.13 ± 0.24	50.13 ± 0.29	64.37 ± 0.34	55.37 ± 0.18	69.05 ± 0.14	58.47 ± 0.54

Table 3
WILCOXON STATISTICAL TEST RESULTS WHEN COMPARED THE TOP SURFACE HARDNESS OF DYRACT EXTRA HEATED AT DIFFERENT TEMPERATURE

Test Statistics ^a						
	T37 - T21	T50 - T21	T60 - T21	T50 - T37	T60 - T37	T60 - T50
Z	-1.214 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b
Asymp. Sig. (2-tailed)	.225	.043	.043	.043	.043	.043

a. Wilcoxon Signed Ranks Test
b. Based on negative ranks.

Table 4
WILCOXON STATISTICAL TEST RESULTS WHEN COMPARED THE TOP SURFACE HARDNESS OF BEAUTIFIL II AND G-AENIAL POSTERIOR HEATED AT DIFFERENT TEMPERATURE

Test Statistics ^a						
	T37 - T21	T50 - T21	T60 - T21	T50 - T37	T60 - T37	T60 - T50
Z	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b
Asymp. Sig. (2-tailed)	.043	.043	.043	.043	.043	.043

a. Wilcoxon Signed Ranks Test
b. Based on negative ranks.

three materials at 60°C, followed by the hardness recorded at 50, 37 and 21°C. For top surfaces, the lowest hardness value was recorded in Dyract eXtra group when samples were warmed at room temperature and the highest hardness value was obtained in Beautifil II group when samples were heated at 60°C. For the bottom surfaces, Dyract eXtra specimens heated at 21°C presented the lowest hardness values and Beautifil II samples heated at 60°C presented the highest hardness values. On top and on bottom surfaces Dyract eXtra showed the lowest hardness values, followed by G-aenial Posterior and Beautifil II, irrespective the heating temperature.

Significantly statistical results were obtained when compared the top surface hardness of Dyract eXtra, Beautifil II and G-aenial Posterior after heating at different

temperature, except Dyract eXtra mean hardness at 21°C and 37°C (tables 3 and 4).

When compared the bottom hardness values, significantly statistical results were obtained for each material when the materials were heated at 21, 37, 50 and 60°C, except the bottom hardness of Dyract eXtra and Beautifil II heated at 21 and 37°C (tables 5, and 6).

The mean microhardness ratio of the tested resin-based materials pre-heated at different temperatures is presented in table 7. At 21°C the lowest hardness ratio was recorded for Dyract eXtra, followed by Beautifil II and G-aenial Posterior. Similar ratio results were obtained for Beautifil II and Dyract eXtra when the materials were heated at 37°C. At 37°C, G-aenial Posterior presented higher hardness ratio than Beautifil II and Dyract eXtra. When

Table 5
WILCOXON STATISTICAL TEST RESULTS WHEN COMPARED THE BOTTOM SURFACE HARDNESS OF DYRACT EXTRA AND G-AENIAL POSTERIOR HEATED AT DIFFERENT TEMPERATURE

Test Statistics ^a						
	B37 - B21	B50 - B21	B60 - B21	B50 - B37	B60 - B37	B60 - B50
Z	-1.753 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b
Asymp. Sig. (2-tailed)	.080	.043	.043	.043	.043	.043

a. Wilcoxon Signed Ranks Test
b. Based on negative ranks.

Table 6
WILCOXON STATISTICAL TEST RESULTS WHEN COMPARED THE BOTTOM SURFACE HARDNESS OF BEAUTIFIL II HEATED AT DIFFERENT TEMPERATURE

Test Statistics ^a						
	B37 - B21	B50 - B21	B60 - B21	B50 - B37	B60 - B37	B60 - B50
Z	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b	-2.023 ^b
Asymp. Sig. (2-tailed)	.043	.043	.043	.043	.043	.043

a. Wilcoxon Signed Ranks Test
b. Based on negative ranks.

	21°C	37°C	50°C	60°C
Dyract eXtra	0.80	0.82	0.75	0.72
Beautifil II	0.83	0.82	0.84	0.81
G-aenial Posterior	0.85	0.83	0.85	0.84

Table 7
MEAN MICROHARDNESS RATIO OF THE TESTED MATERIALS

Table 8
WILCOXON STATISTICAL TEST RESULTS WHEN COMPARED THE RATIO HARDNESS OF DYRACT EXTRA HEATED AT DIFFERENT TEMPERATURE

Test Statistics ^a						
	R37 – R21	R50-R21	R60-R21	R37 – R50	R37 – R60	R50 – R60
Z	-1.604 ^b	-2.023 ^c	-2.023 ^c	-2.032 ^c	-2.032 ^c	-2.032 ^c
Asymp. Sig. (2-tailed)	.109	.043	.043	.042	.042	.042

a. Wilcoxon Signed Ranks Test
b. Based on positive ranks.
c. Based on negative ranks.

Table 9
WILCOXON STATISTICAL TEST RESULTS WHEN COMPARED THE RATIO HARDNESS OF BEAUTIFIL II HEATED AT DIFFERENT TEMPERATURE

Test Statistics ^a						
	R37 – R21	R50-R21	R60-R21	R37 – R50	R37 – R60	R50 – R60
Z	-.272 ^b	-.406 ^c	-1.214 ^b	-.962 ^c	-1.633 ^b	-1.841 ^b
Asymp. Sig. (2-tailed)	.785	.684	.225	.336	.102	.066

a. Wilcoxon Signed Ranks Test
b. Based on negative ranks.
c. Based on positive ranks.

Table 10
WILCOXON STATISTICAL TEST RESULTS WHEN COMPARED THE RATIO HARDNESS OF G-AENIAL POSTERIOR HEATED AT DIFFERENT TEMPERATURE

Test Statistics ^a						
	R37 – R21	R50-R21	R60-R21	R37 – R50	R37 – R60	R50 – R60
Z	-2.023 ^b	-1.732 ^c	-1.857 ^b	-2.032 ^c	-1.841 ^c	-2.041 ^b
Asymp. Sig. (2-tailed)	.043	.083	.063	.042	.066	.068

a. Wilcoxon Signed Ranks Test
b. Based on negative ranks.
c. Based on positive ranks.

heated at 50 and 60°C, G-aenial Posterior presented the highest hardness ratio followed by Beautifil II and Dyract eXtra.

Statistically significant results were obtained when compared the hardness ratio at different temperature for Dyract eXtra, except the ratio when material was heated at 21°C and 37°C (table 8). For Beutifil II no significantly statistical results were obtained when compared the hardness ratio of the samples heated at 21, 37, 50, and 60°C (table 9). For G-aenial posterior statistically significant results were obtained when compared the hardness ratio when the material was heated at 37°C to the hardness ratio when the material was heated at 21°C, when the material was heated at 37°C to the hardness ratio when the material was heated at 50°C (table 10).

Previous studies demonstrated that variables like pre-heating temperature, composite type and composition, top or bottom surfaces can significantly influence material hardness [10, 26]. In our study warming the materials at 21, 37, 50, and 60°C led to higher surface hardness (top and bottom) for all type of resin-based tested materials. Similar results were found in studies that evaluate only the composite resins surface hardness in relation with preheated temperature [26]. It was established that surface hardness evaluation is an indirect method to determine the conversion rate of composite resins. Direct positive relation was established between the hardness and the degree of conversion [27, 28]. Increased degree of

conversion (6-18%) was reported in previous studies when composite resins were pre-heated up to 54°C [29]. Similar results were also obtained by Daronch et al. [9], which showed that the degree of conversion was significantly influenced by pre-heated on top and bottom of composite resins. Previous studies showed that a hardness ratio of 80% can be related to a bottom-to-top conversion of 90%, but the correlation between these two parameters was not very accurate [28]. Also, studies have demonstrated that the rate of conversion is not related to resin-based composite formula [27, 28].

The degree of conversion is in direct relation to the type of composite resin. Dyract eXtra resin matrix is a mixture of several methacrylate resins like ethoxylated Bisphenol-A-dimethacrylate, urethane resin, triethylene glycol dimethacrylate (TEGDMA), and trimethylolpropane trimethacrylate (TMPTMA). TCB resin is also included to give the resin mixture a high cohesion, to reduce the hydrofobic characteristics, and to increase the rate of fluoride release. Base resin of Beautifil II is represented by Bis-GMA (7.5 wt%) and TEGDMA (5 wt%). Resin matrix of G-aenial Posterior consists of a mixture of urethane dimethacrylate (UDMA) and dimethacrylate co-monomers and is bis-GMA free. Previous studies showed that TEGDMA presented the highest degree of conversion when compared to other resin composite monomers (Gajewski). That could be an explanation for better microhardness

results obtained for Beautifil II and Dyract eXtra when compared to G-aenial Posterior.

Increasing the temperature of composite resins might lead to a higher molecular mobility and the propagation stage will need longer time without being diffusion controlled. The temperature below the glass transition will improve the mobility of polymer chain, postponing the reaction diffusion-controlled termination [26, 29]. By improving the monomer conversion, the glass transition temperature will be increased due to increased monomer conversion and will induce a greater amount of conversion at higher polymerization temperatures. For dimethacrylate based composite resin a large increase in polymerization rate will occur at small increase of temperature [8]. It was demonstrated that the higher the degree of conversion, the greater the cross linking and the better the mechanical properties [10, 30].

Another factor that can influence the surface hardness is the filler content [31, 32]. Dyract eXtra filler component is represented by strontium fluoride glass and the mean particle size is of 0.8 μm . Beautifil II is an universal nano-hybrid composite that incorporates S-PRG (surface pre-reacted glass ionomer) technology consisting in stable phase of glass-ionomer (fluoroboroaluminosilicate glass). In the filler structure, discrete nano fillers (10-20 nm) were also added in order to obtain a filler load of 83.3 wt% (68.6 vol%). *Particle size ranges* from 0.01 μm to 4.0 μm , with a *mean particle size* of 0.8 μm . Two different kind of pre-polymerized fillers are used in G-aenial Posterior structure. They are produced by polymerizing a resin matrix in which micro-fillers were incorporated, and then milling the polymerized resin into particles averaging 16 to 17 μm in size. The fillers also consist in fumed silica particles and strontium and lanthanoid fluoride dispersed between the pre-polymerized fillers. Total filler load is 77% by weight and 65% by volume. In our study, irrespective that Beautifil II and Dyract eXtra materials have the same mean particle size, they showed different hardness due to the fact that they present different filler content. Increased filler content of Beautifil II (83.3 wt%) and Dyract eXtra might explain the higher hardness values found for this material when compared to G-aenial Posterior (77 wt%).

In our study increased top and bottom surface hardness was obtained for all three resin-based materials. Top surface hardness was higher than bottom hardness, irrespective of the tested material. One possible explanation is the fact that the activation light is reflected, scattered and absorbed while passing through the material. For composite resins, a 75% reduction of the irradiance was reported at a depth of 2mm when compared to the top surface [33]. Studies reported 50% to 70% conversion of monomers at room temperature [34-36]. It is considered a proper polymerization when the bottom/top surface hardness is 80-90%. In our study, all the materials heated at all tested temperatures had the bottom/top ratio ranking from 80% to 85%, except Dyract eXtra heated at 50°C and 60°C, which showed lower ratio (75% and 72%, respectively). Top surface hardness obtained in the present study were similar to that obtained in previous studies [37].

Conclusions

Warming at 21, 37, 50, and 60°C significantly increases the surface hardness (top and bottom) for tested compomer, giomer and composite resin. In the conditions of this study, all the materials heated at tested temperatures

presented the bottom/top ratio ranking from 80% to 85%, except Dyract eXtra heated at 50 and 60°C, which showed lower ratio.

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