Original Article

Effect of Preheating on the Microhardness of Nanohybrid Resin-based Composites

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ABSTRACT

Purpose- The physical and mechanical properties of resin composites are highly affected by the extent of conversion obtained by polymerization. The hardness test can be used as an indirect method to evaluate degree of conversion.The purpose of this study was to evaluate the effect of three preheating temperatures on microhardness of three different nanohybrid resin based composites.

Methods- The 30 specimens for each commercial resin composite [Grandio (Voco), Simile (Pentron) and Tetric N-Ceram (Ivoclar Vivadent)] were randomly subdivided in 3 subgroups which 10 specimens were used for each one {subgroup 1 = preheating at room temperature 21˚ C, subgroup 2 = preheating temperature 37˚ C and subgroup 3 = preheating temperature 54˚ C}. The specimens were photopolimerized with QTH light-curing unit for 20 s following the preheating process. Vickers microhardness test was performed for the top and bottom surfaces of each specimen. Three random indentations were taken for each surface and a mean value was calculated.

Results- The microhardness values in Grandio group were significantly different between all three subgroups (p value ≤ 0.001). In Simile group the only significant difference was between 21˚ C and 54˚ C (p value ≤ 0.005) and in Tetric N-Ceram group the difference between 21˚ C and 54˚ C (p value ≤ 0.001) and also between 21˚ C and 37˚ C (p value ≤ 0.01) were considered as statistically significant.

Conclusion- Regardless of the resin composite material used, surface hardness was considerably improved by increasing temperature. The microhardness values were influenced significantly by resin-based composite brand.

Keywords:
Preheating,
Microhardness,
Resin-based composite,
Nanohybrid.

1. Introduction

In addition to providing a natural appearance, resin-based composites are used for different applications in modern operative dentistry. Despite significant progress in fabrication of these materials over 50 years ago [1], some drawbacks related to mechanical properties, polymerization shrinkage and induced shrinkage stresses, mismatch in thermal expansion, fracture resistance, and marginal leakage are still remaining [2]. The main structure of resin-based composites consists of an organic resin - based matrix and inorganic fillers which has the ability to undergo additional polymerization [3]. The physical and mechanical properties of resin composites are highly affected by the extent of conversion obtained during polymerization [3, 4]. Degree of conversion is calculated by dividing the amount of reacted C = C double bonds by the total number of C = C bonds which are present in dimethacrylate monomers of polymeric matrix

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[5]. Two high molecular weight monomer which are commonly used in the conventional resin composites are bisphenol- A glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA). A considerable number of double C = C bonds in the resin composites based on these monomers remain unreacted [4]. At room temperature only 50% to 75% conversion of monomers can be achieved [3, 6]. Besides reducing the mechanical strength of restoration, oxidation of unsaturated monomers may give rise to composite color changes as well as allergic reactions. An increase in degree of conversion improves surface hardness, flexural strength and modulus, fracture toughness, diametral tensile strength, and wear resistance [7]. According to some studies, the mobility of both radical and monomer in resin-based composites will be increased by preheating which consequently may result in a higher degree of conversion. One of the problems during the placement of conventional resin composites is their adaptation with the tooth cavity walls to avoid gap formation and provide better seal. It seems to be more important when using highly filled resin composites [8]. The handling of resin composites seems to be facilitated by preheating [6].

Hardness is the resistance of a material against indentation. There is a relationship between hardness, material's strength and proportional limit. In dentistry, hardness shows the ability of a restoration to abrade or to be abraded by opposing structures. As a result, the factors affecting the hardness may influence the durability of the restoration [9]. Some studies have shown a correlation between degree of conversion and hardness [10- 12]. Although there are several direct methods including differential thermal analysis (DTA), infrared spectroscopy (FTIR) and raman spectroscopy, hardness test can be used to evaluate the degree of conversion indirectly [13, 14]. It is also a very useful mechanical test especially in the cases with large areas of masticatory forces [15]. Nanohybrid resin composites have a modified structure of microhybrids with more nanoparticles and possibly pre-polymerized resin fillers. Nanofilled and nanohybrid resin-based composites have the potential to provide both excellent aesthetics and improved mechanical properties [16]. As Nanohybrid resin composites are claimed to have the positive characteristics of macrofilled and microfilled resin composites together, they are widely used as universal resin composites in both anterior and posterior teeth [17].

The purpose of this study was to evaluate the effect of three preheating temperature on the microhardnes of three different nanohybrid resin based composites.

2. Materials and Method

Three commercial nanohybrid resin-based composites [Grandio (Voco, Cuxhaven, Germany), Simile (Jeneric Pentron, Wallingford, CT, USA) and Tetric N-Ceram (Ivoclar Vivadent, Schaan,. Liechtenstein)] in the shade of A2 were used in

Table 1. Composition of tested resin-based composites.

Resin-based composite	Manufacture	Matrix	Filler type and size	Filler content (vol.%)
Grandio	Voco, cuxhaven, Germany	BisGMA, UDMA, TEGDMA, DMA	Glass–ceramic (1µm), SiO_2 (20-60 nm)	71.4%
Simile	Jeneric Pentron, Wallingford, CT, USA	PCBisGMA, BisGMA, UDMA, HDDMA	Barium boro-silicate glass, nanoparticulate silica, zirconium silicate (5-20 nm), Glass–ceramic SiO_2 (0.04-0.7)	68%
Tetric N-Ceram	Ivoclar Vivadent, Schaan, Liechtenstein	BisGMA, UDMA, TEGDMA, EthoxylatedBis-EMA	Barium aluminium silicate glass (0.4 µm, 0.7 µm), ytterbium trifluoride (200 nm), mixed oxide (160 nm), Prepolymer	55–57%

this study. Table 1 shows the compositions of resin composites.

2.1. Specimen's Preparation

To evaluate the efficacy of preheating, a total of 90 Disk-shaped specimens were fabricated in a Teflon mold (10 mm diameter x 2 mm thick) according to manufacturers' instructions which 30 specimens were belonged to each commercial resin-based composite. The 30 specimens for each commercial resin composite were randomly subdivided in 3 subgroups including 10 specimens for each one {subgroup 1= preheating at room temperature 21˚ C, subgroup 2 = preheating temperature 37˚ C and subgroup 3 = preheating temperature 54˚ C}. To ensure the accuracy of exam, all the composites were selected of A2 shade. For the process of preheating, the capsules of the each composite were warmed in a dry and dark laboratory oven at the specified temperatures for 30 minutes. A digital thermometer (Traceable® Platinum Ultra-Accurate Digital Thermometer, Thomas Scientific, Swedesboro, NJUSA) was applied to confirm the precise temperature of oven. To avoidmultiple heating and achieve a homogeneous heating process, unit dose capsule forms of the resin composites were used instead of syringe ones. The time taken to move the capsule from the oven and initiate the specimen preparation was approximately 30 seconds. For the preparation of specimens the mold was placed on mylar strip on a glass slab and then was filled with resin composite and packed with a proper condenser under low light conditions. Subsequently, the resin composite was covered with another mylar strip and pressed with a glass slide to extrude excess material [18]. The specimen was light-cured in close contact with its surface through the top mylar strip. Then the specimen was immediately photopolymerized with QTH (Coltolux ® 75-Germany) light-curing unit for 20 s. The light intensity of light-curing unit was measured with radiometer (Optilux, Model 100, 10503, Kerr, USA), which was over 600 mW/cm^2. The specimens were polished with a sequence of 600, 800 and 1200 grit silicon carbide paper under wet conditions and stored in distilled water [19] in a dark oven at 37 ˚C for 24 h to complete the polymerization process.

2.2. Microhardness Test

Vickers microhardness test was performed for each specimen at the top and bottom surfaces using a microhardness tester (Bareiss Prüfgerätebau GmbH, D-89610 Oberdischingen, Germany) under a 200 gr load and a dwell time of 15 s. Three indentations with the random distance of 1 mm were taken for each surface and a mean value was calculated. The microhardness was determined through the measuring the diameters of indentation which was produced by pyramidal square-base diamond indenter. The mean bottom/top ratio was calculated by dividing VHN of the bottom surface by VHN of the top surface.

2.3. Statistical Analysis

Two and three way ANOVA analysis with independent variables including commercial brand of resin-based composite (three variables), preheating temperature (three variables) and depth of cure (two variables, top and bottom) and Tukey's Post-hoc test with significance level of 95% were performed.

3. Results

Table 2 shows the mean Vickers microhardness values for the top and bottom surfaces of three commercial resin-based composites with three different preheating temperatures. Regardless of the resin composite, surface hardness was considerably increased by increasing composite temperature. The microhardness values in Grandio group were significantly different between all three subgroups (preheating temperature of 21˚ C, 37˚ C and 54˚ C) (p value $\leq$ 0.001). In simile group the only significant difference was between 21˚ C and 54˚ C (p value $\leq$ 0.005) and in Tetric N-Ceram group the difference between 21˚ C and 54˚ C (p value $\leq$ 0.001) and also between 21˚ C and 37˚ C (p value $\leq$ 0.01) were considered as statistically significant. For top surfaces, the highest and lowest microhardness values were observed in Grandio groups with the preheating temperature of 54˚ C (Microhardness: 125/36 VHN) and Tetric N-Ceram groups with the preheating temperature of 21˚ C (Microhardness: 53/0 VHN), respectively. Grandio with the preheating temperature of 54˚ C (Microhardness: 121/81VHN) and Tetric N-ceram with the preheating temperature of 21˚C (Microhardness: 43/71VHN) showed the highest and lowest microhardness values on bottom

surfaces, respectively. The mean values of Vickers microhardness ratio (bottom/top) are presented in Table 3 which the highest value was 97% for Grandio with the preheating temperature of 54˚ Cand the lowest one was 82% for Tetric N-Ceram with the preheating temperature of 21˚ C.

Table 2. The microhardness values of top and bottom surfaces for different preheating temperature.

Composites \ Temperatures		21˚ C		37˚ C		54˚ C	
		Mean (%)	(SD)	Mean (%)	(SD)	Mean (%)	(SD)
Grandio	Top	115.93	4.28	118.8	3.29	125.36	3.89
	Bottom	105.07	6.25	111.84	4.40	121.81	3.56
Simile	Top	67.75	5.17	67.95	5.41	71.09	3.75
	Bottom	59.70	4.53	64.17	5.11	66.11	3.89
Ttric N-Ceram	Top	53.0	2.27	54.4	3.14	56.82	3.33
	Bottom	43.71	2.89	49.9	4.5	51.69	6.18

Table 3. Mean Ratio % (bottom/top) for different preheating temperatures.

Temperatures / Composites	21˚ C	37˚ C	54˚ C
Grandio	90	94	97
Simile	88	94	93
Ttric N-Ceram	82	91	90

4. Discussion

Hardness measurement is an indirect method to evaluate the conversion of carbon double bonds in a resin-based composite. It has been shown that a bottom-to top VHN of 80% is related to a bottom-to-top conversion of 90%. However Bouschlicher *et al* refused an accurate correlation between these two parameters. They also stated that the ratio of bottom to top degree of conversion is independent of resin-based composite formulation [11, 12]. It is well known that there is a relation between polymerization process and temperature changes [3]. Trujillo *et al* stated that warming composite resin within biologically compatible temperatures could improve the rate and conversion of polymerization [20]. According to the findings of the present study, all the variables including composite type, top or bottom surfaces of the specimens and the preheating temperature had significant effect on microhardness values. Microhardness was improved by increasing the temperature from 21˚C to 54˚C (bothon top and bottom surfaces) in all groups. However, it was more noticeable in Grandio group. By rising the temperature from 21˚ C to 54˚ C the bottom to top microhardness ratio was increased. The highest values were observed in the Grandio group with the temperature of 54˚ C (97%) and Tetric N-Ceram with the temperature of 21˚C had the lowest one

(82%). Similarly, Trujillo and Stansburyvia the application of near-infrared spectroscopic technique concluded that preheating up to 54˚ C leads to 6-18% increase in degree of conversion depending on the type of resin composite. They also reported 51% to 92% reduction in curing time to reach an equivalent conversion at room temperature [21]. Daronch *et al* studied the effect of different curing times and preheating temperatures on monomer to polymer conversion. They concluded that the degree of conversion was significantly affected by preheating on both top and bottom surfaces and for all light curing times. They also stated that the resin composites preheated at 54˚ C or 60˚ C with an exposure time of 5 s show more degree of conversion than 40 s exposure time at room temperature [22].

Some factors have been proposed for the increased conversion of preheated composites. An elevated composite temperature leads to an increase in molecular mobility. Therefore, the propagation stage takes longer time without becoming diffusion controlled. Furthermore, temperature rise below the glass transition improves the mobility of polymer chain, postponing the reaction diffusion-controlled termination. By improving the monomer conversion the glass transition temperature will be increased inducing a greater amount of conversion at higher polymerization temperatures. Dimethacrylate based systems show an Arrhenius behavior which means a small increase in temperature shows a large increase in polymerization rate [23]. By improving the degree of conversion, a greater cross linking and as a consequence better mechanical properties will be expected [20, 24]. However, the mechanical properties are dependent on the characteristics of polymer network formation and these are not equivalent to conversion [22]. It is worth mentioning that the depth of cure evaluated in this study was 2 mm which is the maximum accepted thickness for the placement of resin composites. As the shade of composite can influence the hardness values, all the composites were selected of A2 shade which is the most common shade in indirect restorations [25, 26]. Cook and Standish showed that although the elevated temperature may result in a same conversion with shorter irradiation time, the depth of cure will probably be less due to the logarithmic relationship between the depth of cure and irradiation dose [27].

According to the results of this study, the highest microhardness values were obtained for Grandio and Tetric N-Ceram showed the lowest. As shown in some studies, there is a direct relation between filler content and microhardness values [28, 29]. The filler content of Grandio, Simile and Tetric N-Ceram are 71.4%, 68% and 55–57% vol.%, respectively. Unlike Simile, TEG-DMA is used in the formulation of Grandio as diluent monomer. Gajewski *et al* concluded that TEG-DMA has the highest degree of conversion among the monomers used in resin composites [30]. As shown in previous studies, there is a positive relation between degree of conversion and microhardness [11, 12]. Moraes *et al* stated that the higher microhardness values of Grandio are probably related to the large particles and higher filler content [31]. However, Cekic-Nagas *et al* concluded thatthe filler loading and the formulation of organic matrix are more critical factors than the filler particle size [32]. In the present study all the groups showed improvement in the microhardness of both top and bottom surfaces.While passing the composite layers, the light is scattered and absorbed. As a result, the microhardness values on top surfaces are higher than the bottom ones [33].

Increasing the temperature of tooth cavity due to preheated composite may be an issue of concern. When there is 1 mm of remaining dentin, using composite resin preheated up to 130˚ Fleads to only 1.6˚ C increase in pulpal temperature [34]. Regarding to the study of Zach and Cohen the critical threshold for pulp damage is 5.5 ˚ C [35]. Daronch *et al* showed that by heating resin composite to 60˚ C the extent of temperature increase was only 0.8 ˚ C while a 5˚ C intrapulpal temperature rise was seen during light curing process [36]. However, the thickness of remaining dentin is a critical factor in pulpal temperature rise [37]. Another concern is leaving composite resin in heater for an extended time that may lead to premature polymerization. Irrespective of the cure time (20 s or 40 s), resin composite can be heated up to 8 hours without any premature polymerization [34]. Torres *et al* showed that preheating composite up to 54˚ C increases the microhardness on the top surfaces significantly. Composite specimens preheated to 54˚ C didn't show significant difference in microhardness values when irradiated for 20 s or 40 s. They concluded that maximum degree of

conversion on the top surface was obtained at half of the recommended curing time. Microhardness was not affected significantly by ambient (24˚ C) or cooling (5˚ C) temperatures [38]. In several studies it has been shown that preheating composite could improve the polymerization efficacy and mechanical properties of resin composites [8, 39]. However, Saade *et al* concluded that preheating of the resin composites has no effect on Vickers hardness values. They used Tetric Ceram which is a microhybrid resin composite [40].

The handling properties will be improved by preheating uncured resin based composites. Lower viscosity leads to better adaptation to the cavity wall which is especially useful for very stiff composites [20]. To achieve a good marginal contact and minimize the gap between the tooth cavity and the restoration, flowable resin composites have been introduced. However, due to the low filler content of the flowable composite, they show higher shrinkage than the universal composites [3]. There have been some concerns about the probable shrinkage of preheated resin composites. Elhejazi concluded that rising temperature from 23˚ C to 60˚ C may lead to an increased shrinkage from 1.26% up to 2.29% [41]. However, Wagner *et al* showed that preheating composite produces significantly less microleakage at the cervical margin compared to the control or the flowable resin composites. They also stated that delayed curing of preheated composites has adverse effects on microleakage, so it is not recommended [42].

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