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EFFECT OF THERMAL CYCLING ON MICROHARDNESS VALUES OF RESIN COMPOSITES WITH DIRECT AND INDIRECT INDICATIONS CURED BY DIFFERENT POLYMERIZATION TECHNIQUES

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ABSTRACT

Introduction The present *in vitro* study investigated the effects of cyclic temperature changes on the surface hardness of composite resins that could be used indirectly and cured by different polymerization methods.
Methodology A total of 200 disc-shaped samples (5 mm x 2 mm) were prepared from resin composites with indirect indications (ENA HRi Bio Function [BF], Enamel Plus HRi [HRI]), an indirect composite (Gradia Plus [GR]), and a direct composite (Herculite XRV Ultra [HL]). Composite samples were divided into subgroups according to the 20- or 60 seconds polymerization methods with either a light emitting diode (LED), halogen curing units, or a dual mode light curing unit (Labolight DUO) (n=10). Then, the specimens were subjected to ageing through 5000 thermal cycles at temperatures alternating between 5°C and 55°C with a dwelling time of 30 seconds in water baths. Finally, all samples were subjected to hardness testing using a digital microhardness tester. Scores in Vickers values were analyzed statistically using the ANOVA and Bonferroni tests at $p<0.05$.
Results Thermal cycling had significantly affected the microhardness values of groups polymerized with both the halogen (GR- 60 seconds, HRI- 60 seconds) and LED units (HRI- 60 seconds). Dual mode curing had significantly increased the microhardness scores of HRI and HL groups ($p<0.05$). Among all groups, regardless of the curing time or unit, BF had the highest microhardness scores.
Conclusion Although thermal cycling had significantly affected the microhardness scores of some groups of composite resins with indirect indications, its efficiency could be reported as inconsiderable.

KEYWORDS

Dual mode curing; Polymerization; Microhardness; Resin composites; Thermal cycling.

1. INTRODUCTION

Improvements in the materials science and adhesive technology have led dental clinicians to have a wide spectrum of options for tooth-coloured restorations, even when the teeth are severely damaged. In the past, most of these cases were treated with crown restorations, but now, onlays and overlays are highly common and applied frequently. Furthermore, nowadays, there are restorative materials which could be used for both direct and indirect applications of composite resins. These materials are conventional composite resins comprised of increased filler ratios and adapted to extraoral curing settings [1]. With higher filler ratios and increased curing time, these materials are reported to provide higher mechanical properties [2].

Common curing methods used for the composite materials with indirect indications include high energy irradiation, which can be conducted through the use of different polymerization devices, from

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halogen to light emitting diodes (LEDs), or additional devices such as laboratory equipment [1]. After the polymerization reaction of the resin composites occurs, a three-dimensional cross-linked network is developed, which is termed the "polymer chains" [3]. The degree of conversion of the materials could change in all of the polymerization methods, which could consequently lower the final properties at the end. The degree of conversion after the polymerization reactions are previously reported to be up to 77% for direct composite resin materials [4]. Thus, uncured monomers are eluted and able to soften the polymer matrices, resulting in lower microhardness values and a lack of resistance to wear [5]. As the polymerization reaction occurs as same steps as in the halogen and LED curing units, laboratory polymerization devices emit light with either constant or intermittent setting, or by using extra polymerization methods such as vacuum or pressure. Labolight Duo is one of these devices equipped with polywave (blue and violet) LED light sources and speculated to ensure optimal hardening of resin materials with high power outlet and different curing modes. Besides, with the relevant additional polymerization methods, a greater monomer conversion is achieved by having more formation of the polymer chains on the indirect usage of suitable composite materials. Studies have also reported higher mechanical properties, such as superior microhardness values [6,7]. However, additional polymerization devices generally have extended curing times. Prolonged curing time have been reported to increase the degree of conversion and lower the residual monomers thus, improving the properties of composite materials [5].

Properties such as microhardness values of composite resins directly increase with the degree of conversion, which describes the actual rate of the chain reaction [8]. However, in the polymerization process, tensions can arise within the composite structure, possibly leading to degenerations and crack formations [8,9]. As a result of these hazardous situations, the properties of the materials could once again be negatively affected. Thus, microhardness of the restorative resin composites is to be measured after dynamic tests that constitute stresses or tensions during and after prolonged polymerization reactions. Thereby, the clinical behavior of the material may be better understood.

Thermal cycling is one of the most common dynamic test mechanisms used to simulate the temperature changes in the oral environment; thus, it is a useful methos for evaluating the physiological ageing of dental materials [10]. These temperature changes may lead to the contraction or expansion of the restorative materials, and as a result of these changes, resin matrices of the materials may absorb water. Ultimately, the degradation of the polymeric network may increase, or existing cracks in the structure may widen [11]. In each case, the resistance of the material will be reduced. In the literature, bath temperature and a number of cycles have not been standardized, but commonly used bath temperatures for testing dental materials are of 5 °C and 55 °C, with a dwell time of 15-20 seconds [10,11]. Apart from the differences in the methodology design of the studies, thermal-cycling had been previously linked to increased ratio of degree of conversion of monomers and surface roughness [12], reduced microhardness and flexural strength of direct composite materials [13]. On the other hand, there are a few studies investigating the post-thermal outcomes of composite resins with indirect indications [14,15]. Since not only thermal cycling but also thermopolymerization methods such as autoclave or microwave were also investigated in those studies, no clear conclusions could be drawn regarding the thermal effect to microhardness scores of indirect composite materials. However, the alterations between the microhardness scores of the indirect composites were attributed to the polymerization mode of the device used or the monomer matrix type [14].

Since there have been similar monomer types among indirect composites and conventional direct composites, the polymerization mode may be investigated. Therefore, the present study investigated the effect of thermal cycling on the microhardness values of composite resins polymerized with different curing devices in order to find out the optimum materials and polymerization methods that could be applied for severely-damaged teeth. The null hypotheses of the study were that there would be no significant differences (1) after thermal cycling among microhardness values of (2) tested materials (3) those cured with various polymerization methods.

2. MATERIALS AND METHODS

2.1 Sample preparation

A nanohybrid direct composite, a nanohybrid indirect composite, and two composite resins which are indicated for both direct and indirect restorations were tested in the present in vitro study. Compositions and types of materials are presented in Table 1.

Table 1. Compositions and types of tested materials*.

Type of Material	Material	Brand	Lot number	Shade	Filler ratio	Composition
Direct nanohybrid composite	Herculite XRV Ultra [Enamel] [HL]	Kerr	6591129	A2	71% wt., 54% vol.	Bis-GMA, TEGDMA, Bis-EMA, SiO2, Barium silicate glass, Prepolymerized filler with barium silicate glass and silica
Indirect composite	Gradia Plus [GRA]	GC	1304201	Enamel [Heavy Body]	71% wt.	1-5% Bis-GMA, 5-10% TEGDMA, 1-5% UDMA; ceramic filler
Nanohybrid composite	Enamel Plus HRi [HRI]	Micerium	2016007321	Enamel [UE2]	75% wt., 53% vol.	UDMA, Bis-GMA, 1,4-butandioldimethacrylate, nano zirconium oxide [20 nm], glass fillers [1 µm]
Microhybrid composite	Enamel Plus HRi Bio Function [BF]	Micerium	2019008590	Enamel [BF2]	74% wt., 60% vol.	UDMA, TCDDMA, silicone dioxide [0.005-0.05 µm], glass fillers [0.2-3 µm]

**Bis-GMA: bisphenol-A glycidyl dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate, Bis-EMA: ethoxylated bisphenol-A-dimethacrylate, TCDDMA: Tricyclodecane dimethanol dimethacrylate. wt%: weight percentage, vol%: volume percentage.*

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A total of 200 samples were fabricated using a cylindrical metallic mold (5 mm in diameter and 2 mm thick). Each material was inserted into the mold and confined between two opposing transparent matrix strips. A glass microscope slide (1 mm in thickness) was then placed over the mold, and constant pressure was applied to extrude the excess material. Afterward the samples were divided into 5 subgroups according to the polymerization methods (n=10): i) Halogen for 20 seconds (Halo 20), ii) Halogen for 60 seconds (Halo 60), iii) Light emitting diode (LED) for 20 seconds (LED 20), iv) LED for 60 seconds (LED 60), or v) A dual-mode curing device (Dual-mode). Curing times, wavelength of the curing device, and the mode of polymerization unit are given in Table 2.

Table 2. Properties and application of the curing devices.

Curing unit	Name	Irradiance [Mw/cm2]	Wavelength [nm]	Application
Halogen	Lunar [Benlioglu Dental, Turkey]	500	380-500	Samples were polymerized for 20 or 60 seconds in direct contact with glass slide.
LED	D-Light Pro [GC, Japan]	1400	T 385-515	T Samples were polymerized for 20 or 60 seconds in direct contact with glass slide.
Dual-mode	Labolight Duo [GC, Japan]	-	T 380-510	T Samples were polymerized in full mode of the curing device for 3 minutes, after 10 seconds polymerization by abovementioned LED curing unit in direct contact with glass slide.

All samples were then removed from the mold, evaluated for visible surface defects, and kept in distilled water at 37 ±1°C for at least 24 hours. Afterwards, finishing and polishing procedures were performed with a silicone polisher kit (Diatech, Coltene, Switzerland) according to the manufacturers' instructions. Then, all samples were washed under running tap water to remove residuals from the polishing procedures and dried gently with air spray. Finally, all of the samples were stored under light-proof conditions in distilled water at 37 ±1°C for at least 24 hours before testing.

2.2. Microhardness testing and thermal cycling

The surface microhardness of the specimens was measured using a microhardness tester (Schimadzu HMV-G, Kyoto, Japan) under a load of 300 g for 15 seconds. The average value of the three indentations for each sample was taken in terms of Vickers hardness number (VHN) and recorded as the "baseline" score. Then the samples were subjected to thermal cycling (SD Mechatronik Thermocycler, Rosenheim, Germany) of 10.000 cycles using 5°C and 55°C water baths, with a dwelling time of 30 seconds and a resting time of 15 seconds. Following the thermal cycling, samples were air-dried and three indentations were assessed again to reach the "final" microhardness scores. Then, all of the "baseline" and "final" scores were averaged. An illustration of the experimental period is given in Fig. 1.

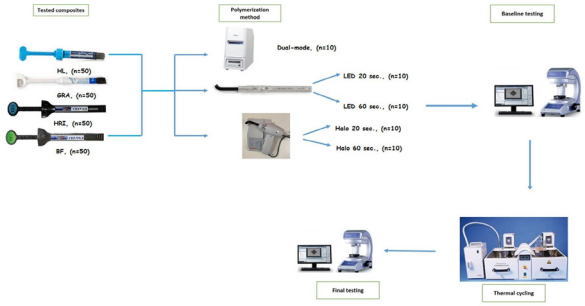

Figure 1. An illustration of the experimental period.

2.3. Statistical analysis

The statistical analysis was done using SPSS 27.0 [SPSS, Chicago, IL, USA] at a significance level of 0.05. The results were primarily analyzed using the Kolmogorov-Smirnov test to determine the existence of a normal distribution. Since the data were normally distributed, differences observed within the baseline and final scores of each material were analyzed by Student's t-test. Further statistical analyses for cross-comparing among groups were performed by the one-way ANOVA and Bonferroni/ Dunn test.

3. RESULTS

Mean microhardness values (VHN) of all groups and statistical analysis within subgroups before (baseline) and after (final) thermal cycling of tested composite resins are given in Table 3.

Table 3. Mean microhardness values with standard (Std.) deviations and the statistical differences within subgroups before and after thermal cycling.

Groups & Subgroups		n	Mean	Std. deviations	p*
HRI-Halo 20	Baseline	10	43.99	2.84	0.28
	Final	10	45.29	3.22	
HRI- Halo 60	Baseline	10	40.13	2.14	0.000
	Final	10	2.93	1.28	
HRI- LED 20	Baseline	10	48.79	2.15	0.27
	Final	10	49.06	1.77	
HRI-LED 60	Baseline	10	46.16	2.22	0.040
	Final	10	48.91	2.28	
HRI-Dual-mode	Baseline	10	57.06	7.60	0.49
	Final	10	56.13	4.55	
BF-Halo 20	Baseline	10	63.13	5.49	0.40
	Final	10	64.27	5.55	
BF- Halo 60	Baseline	10	71.14	3.88	0.34
	Final	10	72.27	3.19	
BF- LED 20	Baseline	10	67.52	7.30	0.68
	Final	10	68.74	3.20	
BF-LED 60	Baseline	10	66.2	3.30	0.10
	Final	10	68.67	2.58	
BF-Dual-mode	Baseline	10	69.31	5.00	0.31
	Final	10	71.01	6.47	

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GRA-Halo 20	Baseline	10	53.91	4.45	0.22
	Final	10	51.35	3.16	
GRA- Halo 60	Baseline	10	48.69	1.87	0.001
	Final	10	50.63	2.31	
GRA- LED 20	Baseline	10	48.91	2.49	0.16
	Final	10	50.82	3.16	
GRA-LED 60	Baseline	10	57.92	5.67	0.43
	Final	10	58.88	3.99	
GRA-Dual-mode	Baseline	10	53.90	3.65	0.51
	Final	10	54.18	1.90	
HL-Halo 20	Baseline	10	43.75	1.66	0.19
	Final	10	45.27	2.19	
HL- Halo 60	Baseline	10	52.73	5.09	0.33
	Final	10	51.30	2.90	
HL- LED 20	Baseline	10	47.97	3.58	0.17
	Final	10	49.26	3.27	
HL-LED 60	Baseline	10	47.90	1.60	0.51
	Final	10	48.96	2.47	
HL-Dual-mode	Baseline	10	4.64	5.62	0.63
	Final	10	54.65	3.30	

*p<0.05 presents significantly different scores within subgroups.

According to the analysis within subgroups regarding the effect of thermal cycling, it could be noted that no significant differences were detected among groups except for subgroups of HRI- Halo 60, HRI-LED 60 and GRA-Halo 60. The final microhardness values of those groups were significantly higher than those of the baseline scores (p<0.05).

The statistical analysis regarding the subgroups of tested materials are given in Table 4.

Table 4. Mean microhardness values of all groups and their significant differences regarding the polymerization methods before and after thermal cycling.*

Materials	Polymerization methods	Baseline	Final
HRI	Halo 20	43.99^{A}	45.29^{a}
	Halo 60	40.13^{A}	42.93^{b}
	LED 20	48.79^{B}	49.06^{a}
	LED 60	46.16^{B}	48.91^{a}
	Dual-mode	57.06^{C}	56.13^{c}
p		**0.000***	**0.000***
BF	Halo 20	63.13^{D}	64.27^{d}
	Halo 60	71.14^{E}	72.27^{e}
	LED 20	$67.52^{D,E}$	$68.74^{d,e}$
	LED 60	$66.25^{D,E}$	$68.67^{d,e}$
	Dual-mode	$69.31^{D,E}$	71.01^{e}
p		**0.015***	**0.003***
GRA	Halo 20	53.91^{F}	51.35^{f}
	Halo 60	48.69^{G}	50.63^{f}
	LED 20	48.91^{G}	50.82^{f}
	LED 60	57.92^{F}	58.88^{g}
	Dual-mode	53.90^{F}	54.18^{f}
p		**0.000***	**0.000***
HL	Halo 20	43.75^{G}	45.27^{g}
	Halo 60	$52.73^{H,I}$	51.30^{h}
	LED 20	$47.97^{G,H}$	49.26^{i}
	LED 60	$47.90^{G,H}$	$48.96^{g,i}$
	Dual-mode	54.64^{I}	54.65^{h}
p		**0.000***	**0.000***

*Uppercase letters of "Baseline" values and lowercase letters of "Final" values of each material group showed significant difference regarding polymerization methods.

Regarding the materials, in most of the polymerization methods, BF showed higher microhardness values among other tested composites. Comparing the only indirect composite resin material, GRA, BF showed significantly higher microhardness values in all polymerization methods (p<0.05). Furthermore, inferior microhardness values were reported for HRI than GRA and HL in groups polymerized with halogen (p<0.05). In LED groups, HRI showed similar microhardness values to HL but had lower scores than GRA. GRA showed significantly higher microhardness values than HL when polymerized with Halo 20 and LED 60.

Among the polymerization methods, the dual-cure curing unit had significantly increased both baseline and final microhardness values of the HRI group only. However, microhardness values of the rest of the composite materials had only improved numerically by dual-cure curing unit. Except for the only indirect composite group of the study, GRA, there were no significant differences among the final microhardness values of the LED groups. In most of the Halo 20 and LED groups, similar microhardness results were gained among tested composites.

4. DISCUSSION

The microhardness of the resin-based composites is directly affected by their polymerization ratio. The prameters regarding light curing of these materials such as exposure time and distance, radiant emittance, and wavelength of the curing unit, could affect the polymerization efficacy [5]. Various polymerization methods may alter the formation rate of double carbon bonds and affect the level of residual monomers in the structure. The efficiency of polymerization could be measured directly by spectroscopic measurements, which are expensive and much more time consuming than indirect methods. On the other hand, micro-hardness is one of the indirect methods and is commonly used in many studies due to its repeatability and simple usage [16]. Thus, the present in vitro study investigated the effect of thermal cycling on the microhardness scores of resin-based composites with indirect applications, which were cured with different polymerization methods.

Composite materials are subjected to temperature and humidity changes in the oral environment which may affect their behavior against upcoming forces and form a physical degradation such as wear, abrasion, and fatigue. These stress conditions are commonly simulated in the laboratory through thermocycling [10,17]. It is one of the most widely accepted methods in the international literature [10], and also allows a transfer

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of the obtained in vitro results to the clinical practice [17]. Aging the materials with thermal cycling may stimulate temperature-related or hydrolytic breakdowns of resin matrices, however, in the present study, there were no considerable changes in the microhardness scores of the tested groups. A significant improvement in the microhardness scores of only three groups (GRA Halo 60, HRI Halo 60 and LED 60) were obtained, so the first hypothesis of the study is partly rejected.

According to the literature, thermal cycling had lowered the microhardness scores [13,18], wear resistance [12], flexural strength [18], and increased the surface roughness [12]. The enhancement in the microhardness scores of the related group of materials, which were polymerized with extended curing time, could be attributed to a couple of reasons. The increased temperature and the thermal shocks provided by the design of thermal cycling increase the monomer movements as well as the elution of unreacted monomers. Some free radicals that are entrapped in the polymerization process were reported to remain in the structure for many weeks [19]. After thermal cycling, these free radicals were able to join within any double bonds and maintain the polymerization reaction, thus leading to a higher final microhardness [13]. Still, the effect of thermal cycling to the microhardness scores of the tested composites may count as inconsiderable.

In both the anterior and posterior areas, one of the main reasons for restoration failures are the fractures of the composite bulk or mix of teeth/restoration structure [20]. So, today's strategies for strengthening the resistance of composite materials are to increase the filler content and improve curing initiation, monomer systems, and polymerization modes [21,22]. With the help of the improvements in the material and adhesion science, there are now various types of composites that could be used when there is a lack of remaining tooth structures. In the present study, direct and indirect composites showed significantly different microhardness scores, thus the second hypothesis is rejected.

GRA as an indirect composite was presumed to show the highest microhardness scores, but in many experimental groups, it is reported to have the second-best microhardness scores. Indirect composites differed from direct composite materials by particular changes in the structure, such as filler and monomer types and/or an improved filler or matrix adhesion [23]. Along with the polymerization methods improved with the laboratory devices such as heat, vacuum, or extra light applications, with some of the previous studies reporting comparable physical and mechanical properties of indirect composites to hybrid ceramics and even CAD/CAM blocks [17,21,23,24].

However, in the present study, the microhybrid composite BF, which has a higher filler ratio (74%) than GRA (71%), showed significantly higher microhardness scores in all groups except for Halo 60. These superior results could not only be attributed to BF's higher filler ratio, but also its monomer content. The bulky three-ring structure of the TCDDMA monomer in BF slows down the polymerization rate and provides more double bonds before the reaction is completed [25].

Thus, the advantageous organic and inorganic contents of BF could be the reason why it has greater microhardness scores in tested polymerization methods.

Moreover, as fillers ratios of the tested materials are similar to each other (between 71% - 75%), the differences in the monomer type may alter the microhardness scores. It was reported that addition of TEGDMA to resin matrices increases the double bond conversion in polymerization reactions [26]. In the present study, the two tested composites involving TEGDMA (GRA and HL) showed similar microhardness values following BF. The related monomer may have an effect on these results, however, its ratio in the structure and the interactions with other monomers may also have an influence on the final microhardness parameter. On the other hand, except for the polymerization with the dual mode curing unit, tested HRI groups showed significantly and numerically lower microhardness scores than HL groups. As HL has both the direct and indirect indications, it is reliable to use with the dual mode curing devices. Although BF and HRI are composites of the same brand and BF showed significantly higher results, it could be erroneous to lead an indisputable opinion on these composite materials. In order to form an indisputable opinion, more of the mechanical tests should be conducted, such as elasticity modulus, and flexural and tensile strength.

The enhancement of a composite material's microhardness after polymerization with various light curing units or methods has been reported in previous studies [16,27,28]. In addition to this, extended curing durations has been investigated with various curing devices in the present study. As the curing time recommended by manufacturers is generally the minimum seconds required, it may not be effective in every clinical setting [29]. Besides, extended curing time may improve the performance of composite materials against oral forces [17]. Regarding the results, there are significant differences between the various polymerization methods tested in the current study, thus, the third hypothesis is also rejected.

Polymerization of composite materials is influenced by many parameters, such as curing time, irradiance of the curing unit, and composite layer thickness [16]. The amount of light transmitted from the surface to the bottom of the composite layer is one of the main specifying factors for final microhardness score [16,29]. The effect of structural differences of the tested materials is widely discussed above, but there is one important point which could affect the light transmittance. In parallel with other studies, "shades" of the composites are chosen to the ones commonly used at the top layer of the restorations [28,29]. So, they were all "enamel" composites that should have the highest transparency than "dentin" or "body" composites.

Extended curing time (60 seconds) improved the microhardness scores of HL, BF, and GRA significantly. The only indirect composite of the current study, GRA, showed significantly different microhardness values in LED groups, however BF and HL also showed significantly different microhardness values in halogen groups. On the other hand, the dual mode device had significantly and numerically improved the microhardness levels, and even the highest values were obtained in two of the groups (HRI and HL). Even though the LED device had a higher light density, the various differences in the scores between LED and halogen groups may be attributed to the materials' compatibility to the

wavelength of the curing devices or the differences between the shade and level of the photoinitiators. The dual mode device used in the present study involves 12 blue and three purple LEDs, thus, as compared to the other devices, the highest light intensity was expected. Although polymerization efficiency is multifactorial, it could still be concluded that all of the tested composite materials had benefited from the wide wavelength spectrum of the dual mode curing device. This result is also in accordance with Mayinger et al. (2021)'s study [1]. Therefore, when compared with the material selection, the polymerization procedure (LED or halogen) may play a minor role. Still, further studies are needed to investigate this hypothesis.

The results of the present in vitro study have to be evaluated in regards to its limitations. There was a limited number of tested samples and a lack of elements of oral cavity. Within the present methodology, only the thermo-hydrolytic effect of the materials was tested, but the influence of extrinsic factors such as beverages, toothpaste, or mouthwashes and also of the intrinsic factors such as masticatory or chewing forces were not included in the present study. Furthermore, in order to highlight the effect of thermal cycling and various polymerization methods, only one example of a parameter, microhardness, was evaluated.

5. CONCLUSION

Various polymerization methods generated by extended light curing and different curing units were investigated in the current study but, thermal cycling did not significantly affect the microhardness scores, so its influence could be counted as inconsiderable. As the manufacturers recommended definite curing times with various polymerization devices, it should be noted that dual-mode curing had significantly improved the microhardness scores of the tested nanohybrid composites only. Thus, various polymerizaton methods and extended curing times tested in the study had affected the microhardness scores depending on the material-basis.

AUTHOR CONTRIBUTIONS

EMM designed the paper; OG and EMM searched the specialized literature; EMM and GS prepared the samples for the experimental part; EM and KE contributed to the thermalcycling and microhardness evaluation; YI analyzed the statistical data; all of the authors contributed to writing the manuscript; SHS critically revised and edited the paper for the final approval of the final version.

CONFLICT OF INTEREST

Authors declare that there is no conflict of interests.

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Questions

1. The level of degree of conversion of direct resin composites are reported to be up to a …… ratio?

❑a. 70%;
❑b. 75%;
❑c. 77%;
❑d. 90%.

2. Which of the following is not a consequence of remaining uncured monomers?

❑a. A reduction in microhardness;
❑b. An increase in microhardness;
❑c. A reduction in wear resistance;
❑d. Organic matrix softening.

3. Which of the following is not a property of laboratory polimerization devices?

❑a. High power outlet;
❑b. Extended curing times;
❑c. Various curing modes;
❑d. Low battery level.

4. Which of the following has not been previously linked with thermal cycling procedures?

❑a. A rise in surface roughness;
❑b A rise in the degree of conversion;
❑c. A rise in surface corrosion;
❑d. A reduction in microhardness.